



**INTERPRETING ASPHALT BINDER MULTIPLE STRESS CREEP AND  
RECOVERY PROPERTIES USING VISCOELASTIC RHEOLOGICAL  
MODELS**

**By**

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## **DECLARATION**

I hereby declare that this thesis entitled “**Interpreting Asphalt Binder Multiple Stress Creep and Recovery Properties using Viscoelastic Rheological Models**” was composed by myself, with the guidance of my advisor, that the work contained herein is my own except where explicitly stated otherwise in the text, and that this work has not been submitted, in whole or in part, for any other degree or professional qualification.

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## ABSTRACT

This paper investigates the intermediate and high temperature rheological properties of asphalt binder on Dynamic Shear Rheometer (DSR) using Multiple Stress Creep and Recovery (MSCR) test. For comparison, intermediate and high temperature measurements were also carried out using Frequency Sweep Test (FST). Evaluation of two unmodified asphalt binder is presented: 40/50 and 60/70 pen grade. In general, the 40/50 pen binder resulted in higher viscoelastic properties and resulted in higher resistance to shear deformation as compared to 60/70 pen. For the binders evaluated, the 100 Pa shear stress level in MSCR test was chosen because it is within the Linear Viscoelastic (LVE) range and therefore the measurements at this stress was used to characterize the fundamental viscoelastic properties of asphalt binders. The four element Burgers model was utilized to estimate the storage and loss modulus values. Statistical analysis was conducted to objectively evaluate the accuracy of the estimated viscoelastic properties. The estimated shear strain value satisfactorily matches the measure one. The Burgers model parameters were found to be suitable and adequate for describing the creep and recovery viscoelastic properties of the asphalt binders included in the study. The shear modulus master curves derived from MSCR and FST test methods were compared and remarkably good agreement was obtained. Overall, the viscoelastic properties of asphalt binders can be captured on the basis of MSCR measurements within the LVE range. The MSCR test method is a simple, quick, and economical test method as compared to the FST test method for characterizing the creep recovery elastic and viscoelastic properties of asphalt binders.

**KEYWORDS:** Asphalt binder, Rheology, creep and recovery, Burger model, and master curve

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## **LIST OF ABBREVIATIONS**

AASHTO	American Association of State Highway and Transportation Officials
AR	Aged Residue
ASTM	American Society of Testing and Materials
AST	Amplitude Sweep Test
DSR	Dynamic Shear Rheometer
FHWA	Federal Highway Agency
FST	Frequency Sweep Test
LVE	Linear Viscoelastic
MSCR	Multiple Stress Creep and Recovery
PAV	Pressure Aging Vessel
PG	Performance Grade
RTFO	Rolling Thin Film Oven
SHRP	Strategic Highway Research Program
SuperPave	Superior Performing Pavement
TTS	Time Temperature Superposition
VE	Viscoelastic
WLF	Williams-Landel-Ferry

# CHAPTER 1

## 1. INTRODUCTION

### 1.1. Background

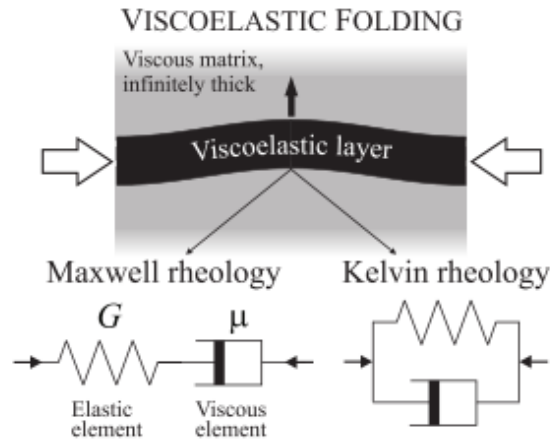
Asphalt binders exhibited both elastic and viscous properties and therefore are called viscoelastic materials. The properties of asphalt binders have traditionally been evaluated using conventional tests as part of the penetration and viscosity graded specification but don't address specific distress mode or ensure long term pavement performance. The latest SuperPave Performance Graded (PG) graded specification is based on the idea that asphalt binder's properties should be related to the conditions under which it is used. The rheological behavior of asphalt binder depends on temperature, time of loading, and aging.

Linear viscoelastic rheological properties of asphalt binder are measured from a device known as the Dynamic Shear Rheometer (DSR) which is one of the tests used in SuperPave PG specifications. The linear viscoelastic (LVE) region will be determined using strains (amplitude) sweep test by applying a varying increasing strain to the sample and observing the resulting stress or modulus. To determine the viscoelastic properties of asphalt binders further, Frequency Sweep Test (FST) will be performed for intermediate to high temperatures and two viscoelastic parameters will be measured: complex modulus ( $G^*$ ) and phase angle ( $\delta$ ).  $G^*$  is the measure of resistance to deformation and  $\delta$  is the measure of the resistance elastic and viscous components. Storage modulus ( $G'$ ) and loss modulus ( $G''$ ) are the two components of the complex modulus.  $G'$  and  $G''$  are the measure of the stored and dissipated energy when load is applied and released. However, when slow moving traffic is simulated under high pavement service temperature, oscillatory rheological tests may not be fully suitable to capture the viscoelastic properties of modified asphalt binders.

The latest improvement to the SuperPave PG specification is the Multiple Stress Creep and Recovery (MSCR) test which provides testing the asphalt binder at high temperature and is blind to modification. This test method also eliminates the need for various Performance Graded (PG) plus tests. The test is conducted on DSR equipment using a residue from Rolling Thin Film Oven (RTFO) which simulates the short term aging of the binder by applying 100 Pa followed by 3200 Pa shear stress level. At each stress level ten cycles will be applied by applying a load for 1 sec and let it recover for 9 sec. Two parameters will be obtained from the test: the Non-Recoverable Creep Compliance ( $J_{nr}$ ) and Percent Recovery (R).  $J_{nr}$  is the measure of the permanent deformation whereas R is the measure of the elastic response of the asphalt binder under repeated creep loading. One of the key benefits of the MSCR test is to simulate the actual field conditions through traffic/loading as well as environmental climatic conditions.

Asphalt binder exhibit viscoelastic properties. The main feature of the elastic behavior is to fully store energy during loading (i.e. creep) and completely dissipate it during unloading (i.e. recovery). Viscoelastic rheological parameters derived from the creep and recovery test can be used to characterize the stiffness and elastic recovery properties. Viscoelastic parameter determination involves describing the creep and recovery test with the use of viscoelastic rheological models. Viscoelastic models can store and dissipate energy at varying intensities using creep and recovery testing. For quantifying the deformation and viscoelastic properties of asphalt binders, the four element Burger's model is widely used. The four element Burger's model comprises a combination of Maxwell and Kelvin/Voigt model connected in series. The Maxwell model consists of a spring and a dashpot connected in series whereas the Kelvin/Voigt model consisting of the association in parallel of the spring and dashpot. A limited number of

studies are currently available evaluating the viscoelastic properties of asphalt binder utilizing creep and recovery measurements.



**Figure 1.** *Maxwell and Kelvin model [14]*

The main objective of this research paper is to experimentally evaluate the viscoelastic properties of asphalt binders using the MSCR test for intermediate and high temperature by utilizing the four element Burger's model. Two unmodified asphalt binder will be investigated. Intermediate and high temperature measurements are also performed using FST to validate the viscoelastic properties of evaluated through MSCR test. The four element Burger's model is will be utilized to estimate the storage and loss modulus values and intern utilized to determine the overall viscoelastic properties of asphalt binders. The correlation between the viscoelastic properties of measured from FST and modeled using the MSCR test results will be examined. The study will present a comparison of shear modulus master curves derived from MSCR and FST test methods. Comparison of the two asphalt binders viscoelastic properties will also be discussed.

## 1.2. Problem Statement

Currently FST and MSCR tests are conducted in the laboratory to determine the viscoelastic properties of asphalt binder at intermediate and high temperature which is time consuming and also not economical. This study focuses on minimizing the test methods. It is proved in many

researches that the FST can't accurately determine the high temperature asphalt binder viscoelastic properties before the introduction of the MSCR test. Therefore this research paper will examine if the overall viscoelastic properties of asphalt binders can be captured on the basis of MSCR measurements at intermediate and high temperature within the LVE range.

### **1.3. Objective**

#### **1.3.1. General Objective**

The objective of this research paper is to estimate the viscoelastic properties of asphalt binders using viscoelastic rheological models.

#### **1.3.2. Specific Objectives**

The specific objectives are:

1. To characterize the fundamental viscoelastic rheological properties of the asphalt binders captured through FST and MSCR Test.
2. To compare the measured and modelled shear strain values of the two asphalt binders.
3. To compare the Viscoelastic (VE) rheological properties of the two asphalt binders captured through MSCR and FST test.
4. To compare the shear modulus master curve derived from FST and MSCR test methods.

### **1.4. Significance**

Due to increased traffic loading, asphalt binders are being modified to improve pavement performance such as resistance to fatigue cracking and permanent deformation. The rheological properties of these modified asphalt binders can be tested using the MSCR test which can simulate the real condition in the field. This allows the Ethiopian highway agencies to use wide variety of modifiers to improve the pavement performance. The direct impact of this research is



to reduce the time and cost spent in the laboratory, longer pavement service life, improved performance and performed quicker than the previous test methods. The other benefit is that, instead of obtaining two parameters from MSCR test we can get the overall viscoelastic properties of the asphalt binder.

## **1.5. Limitations**

The limitations that were encountered while performing this research are:

- 1) The DSR equipment that was available during that time were in Addis Ababa institute of Technology so there were difficulties to get access into the laboratory.
- 2) The asphalt binder that are used in this research are limited to two because other binders were not available in the company I have asked.

## **1.6. Organization**

The study is organized into 5 chapters including this “Introduction” chapter. Chapter 2 presents a literature review focused on definition of asphalt binder and its composition, physical and rheological properties of asphalt binder, viscoelastic nature of the asphalt binder, different grading systems, and the viscoelastic rheological models. Chapter 3 focuses on the different types of asphalt binders used in this study, methodologies, conventional and Superpave tests conducted on the selected asphalt binders. In chapter 4, analysis of the conventional and rheological test results, estimation of burger model parameters, statistical analysis of estimated strain, and comparison of the FST & MSCR master curves are elaborated in detail. Chapter 5 contains conclusions of the study and recommendations for future work. There are appendices in this research, namely A, B, C, D, E, F and G. Appendix A contains conventional test results. The PG test results are presented in Appendix B. Appendix C contains creep and recovery test results. Appendix D elaborates the Burger’s Model parameters test results. The measured and

estimated test results are presented in Appendix E. Appendix F presents the measured and estimated creep and recovery properties. Appendix G contains viscoelastic properties of the tested binders.

## **CHAPTER 2**

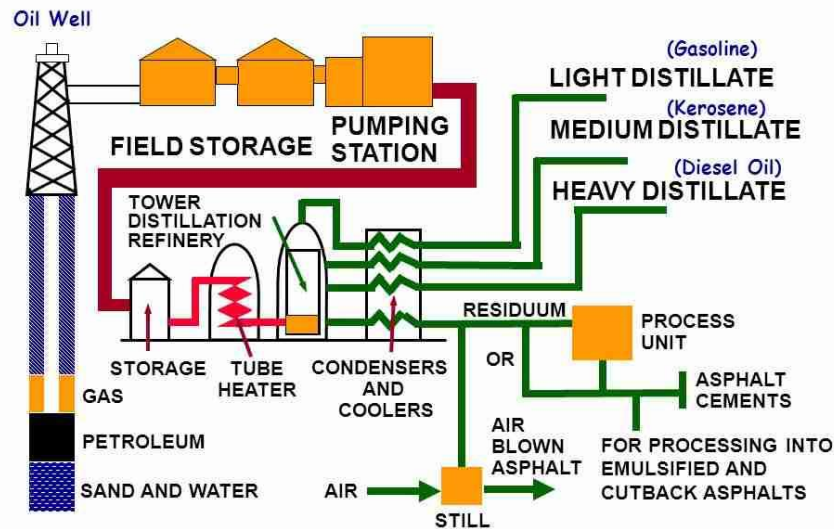
### **2. LITERATURE REVIEW**

#### **2.1. Introduction to Asphalt Binders**

The American Society of Testing and Materials (ASTM) defines asphalt as “a dark brown to black cementitious material in which the predominating constituents are bitumen which occur in nature and are obtained in petroleum processing”. This material is more appropriately called ‘asphalt binder’ or ‘bitumen’ [15]. Asphalt binder has been developed in more recent terminology under the auspices of the United States Strategic Highway Research Program (SHRP) to include modified asphalt cements, unmodified asphalt cements, asphalt emulsions, and asphalt cutbacks. The term “asphalt binder” has been selected to more specifically describe the asphalt material and any other modifiers or ingredients. The terms “asphalt”, “asphalt cement”, “bitumen”, and “asphalt binder” may be used interchangeably, with asphalt cement and bitumen referring more specifically to their petroleum origins and asphalt binder referring to the asphalt cement and any other added ingredient that provides the engineering adhesive used in asphalt pavements [16].

In the late Nineteenth century in Paris, London and United States compacted bituminous pavements were constructed, first of its kind. All Asphalt Binder available was natural at that time. At the beginning of the Twentieth century, Asphalt Binder started being commercially manufactured by refining crude petroleum oil in USA. The need for dust free, smooth, all weather road with the advent of motorized vehicles was felt for the reason, bituminous roads became a worldwide need and also popular. In fact, before used as the principal binder in constructing highways, it was used in roofing, flooring, bridge, sidewalk surfacing, waterproofing etc. purposes.

## Source of Asphalt Cement



**Figure 2.** *Source of asphalt cement [17]*

Asphalt binder at normal atmospheric (ambient) temperatures is a black, sticky, semi-solid, highly viscous, cementitious material. It is typically a solid to semi-solid at normal air temperatures and becomes a liquid at high temperatures. Asphalt is made up largely of a hydrocarbon called bitumen and therefore is often called a bituminous material. Because asphalt binder is sticky, it adheres to aggregate particles and can be used to cement or bind the aggregate in an asphalt concrete mixture. Asphalt binder is an excellent waterproofing material and is unaffected by most acids, alkalis, and salts. This unique combination of characteristics and properties is a fundamental reason why asphalt is an important paving material [18].

Asphalt binder is readily adhesive, highly waterproof, and durable which makes it a valuable engineering material. It imparts a degree of flexibility to mixtures of mineral aggregates due to its plastic (i.e. viscoelastic) nature. Naturally occurring lake asphalt and rock asphalt were used in ancient times as road building and waterproofing material. However, it wasn't until the early 1900's that bitumen became a widespread ingredient in paving material. Around this time

modern petroleum refining techniques were developed which allowed asphalt binders to be manufactured at oil refineries from the distillation of crudes [15].



**Figure 3.** *Trinidad Lake Asphalt* [8]

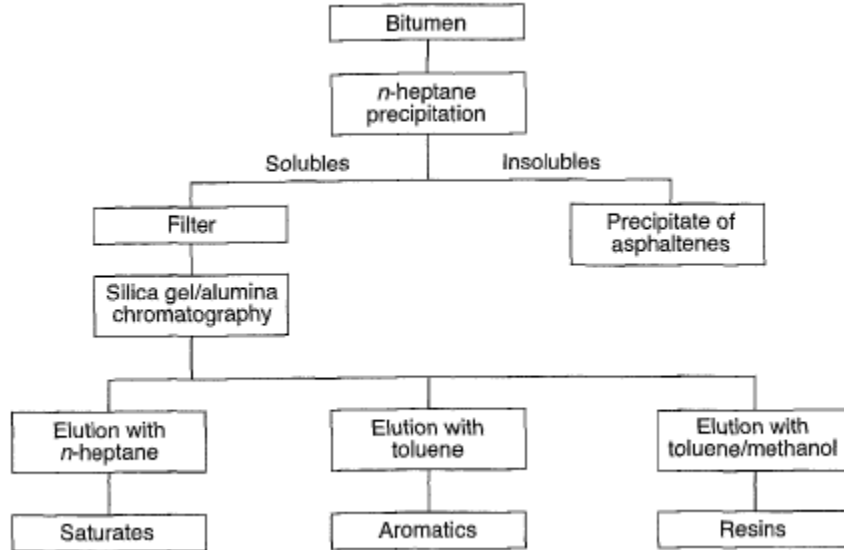
## **2.2. Chemical Constitution of Asphalt binder**

The configuration of the internal structure of asphalt binder is largely determined by the chemical constitution of the molecular species present. It is a complex chemical mixture of molecules that are predominantly hydrocarbons with a small amount of structurally analogous heterocyclic species and functional groups containing Sulfur, Nitrogen and Oxygen atoms. Asphalt binder also contains trace quantities of metals such as Vanadium, Nickel, Iron, Magnesium and Calcium, which occur in the form of inorganic salts and oxides or in porphyrine structures. Elementary analysis of asphalt binder manufactured from a variety of crude oils shows that most asphalt binders contain [19]:

- Carbon 82-88%
- Hydrogen 8-11 %
- Sulfur 0-6%

- Oxygen 0-1.5 %
- Nitrogen 0-1 %

Asphalt binder obtained from distillation of crude oil is a flexible material with a density of  $1\text{g/cm}^3$  at room temperature. But at low temperatures it becomes brittle and high temperatures flows like a viscous liquid. The physical, mechanical and rheological properties of the asphalt binder primarily depend on its colloidal structure, linked to the chemical composition especially to the proportion of asphaltenes and maltenes. Asphaltenes are polar materials of high molecular weight (10,000 to 100,000) that are insoluble in *n*-heptane, a non-polar solvent, and is the straight chain alkane with chemical formula  $\text{H}_3\text{C}(\text{CH}_2)_5\text{CH}_3$  or  $\text{C}_7\text{H}_{16}$  and constitutes 5% to 25% of the bitumen. Maltenes are constituted by resins, aromatic and saturated oils that are soluble in *n*-heptane and possess low molecular weight [20].



**Figure 4.** Schematic representation of broad chemical composition of asphalt binder[19]

### **2.3. Physical Properties of Asphalt Binder**

Asphalt binder is available in variety of types and grades. Asphalt binders are most commonly characterized by their physical properties rather than its chemical properties. For engineering and construction purposes, normally three physical properties of Asphalt binder are important.

#### **2.3.1. Consistency**

Consistency is the term used to measure its degree of stiffness ability to flow. Asphalt binder is thermoplastic material which means it liquefy when heated and solidify when cooled and its state of solidness (stiffness) or liquidness (i.e. ability to flow) is very much temperature sensitive. Consistency of asphalt binder can be judged by some empirical tests such as penetration, softening point, ductility, etc and also by testing the fundamental property of asphalt binder such as viscosity [21].

#### **2.3.2. Purity**

By definition, bitumen is entirely soluble in trichloroethylene. Nowadays, almost entire bitumen is obtained by refining petroleum crude, which are usually more than 99.5% soluble in trichloroethylene. This test is carried out to check the presence of organic materials and impurities in bitumen.

#### **2.3.3. Safety**

Asphalt binder, if heated to a high enough temperature, releases fumes that flash in the presence of a spark or open flame. The temperature at which this occurs is called the Flash point and is well above the temperatures normally used in paving operations. However, to be certain of an adequate margin of safety, the flash point of the asphalt is measured and controlled [22].

## **2.4. Rheology of Asphalt binders**

The word rheology is derived from the Greek word rheo, which translates literally as "to flow". It is the science that deals with the flow and deformation of matter. The rheological characteristics of asphalt binder at a particular temperature are determined by both the constitution (chemical composition) and the structure (physical arrangement) of the molecules in the material. The properties of rheological materials are also time and temperature dependent; consequently, both the time of loading and the temperature of loading must be considered when characterizing the flow properties of rheological materials such as asphalt binders [10]. The rheological properties of asphalt binder are an essential component in the design and production of asphalt pavements. Firstly, the binder must possess enough rigidity to support traffic loads and maintain its shape throughout its service life. However, it must not be so rigid that it cracks at low temperatures or fractures under high levels of stress. Moreover, the binder needs to retain a certain level of fluidity at higher temperatures so that good mixing and compaction are attainable during construction. As a result, the consistency of asphalt binder is of great significance to those in the pavement industry.

The consistency of asphalt binder varies with its chemical composition, which can be linked to its place of origin. Some asphalts are naturally occurring and vary with the asphalt lakes from which they are excavated; their composition is affected by the organic matter that coexists in these lakes. However, most asphalts used in pavements are waste products from petroleum refinement. These asphalts vary in molecular structure based on the crude oil from which they were refined. Because binder composition is so inconsistent, its behavior is extremely variable as well. Therefore, it is important to quantify a binder's fluidity; this is commonly achieved by determining its viscosity [23].



## **2.5. The Viscoelastic Nature of Asphalt Binders**

In order to predict the engineering performance of any material, it is necessary to understand its stress-strain behavior. To determine how a given material will respond to an applied load, laboratory tests must be performed and analyzed, and the results summarized in a form that is readily applicable to engineering design methods. To characterize the stress-strain behavior of materials in the laboratory, the simplest test methods are uniaxial (extensional) tests and shear tests. Such tests may be conducted under controlled stress or controlled strain conditions. The resulting response may then be stated in various ways, depending on the response of the material. Materials for which the stress-strain behavior is linear, and largely independent of time and temperature, can be effectively characterized by the elastic (Young's) modulus. Newtonian fluids, on the other hand, can be characterized through the coefficient of viscosity. Materials such as asphalt binders, which exhibit aspects of both elastic and viscous behavior, are called viscoelastic, and must be characterized with test methods and analytical techniques that account for the time (or rate) of loading and the loading temperature [10].

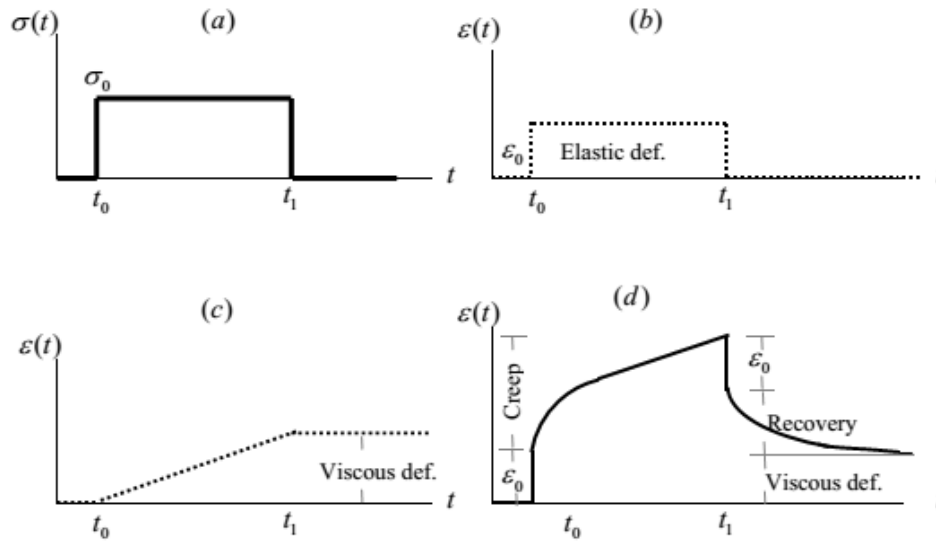
The larger part of asphalt binder consists of carbon and hydrogen, which form non-polar molecules with weak (dispersion) forces of attraction. However, the presence of highly electronegative atoms such as nitrogen, sulfur, and oxygen result in polar molecules with strong attractive forces. The polar and non-polar molecules exist together in a homogeneous mixture in which the polar molecules form a network or structure, and the non-polar molecules form a body of material around the network. The chemical bonds holding the molecules together are relatively weak and can easily be broken by heat or shear stress. This results in the viscoelastic nature of asphalt binder. The polar molecules give asphalt its elastic properties while the non-polar molecules contribute to the viscous properties of the asphalt binder. Due to their similar

physical properties and viscoelastic nature, asphalt binders are often classified with polymeric, or macro-molecular, substances although their chemical composition is significantly different [15].

Asphalt binder's characteristics are dependent on time and temperature because of its viscoelastic property. With higher temperatures and longer loading times, the asphalt binder becomes softer and behaves more like a viscous fluid. With low temperatures and fast loads, the asphalt binder becomes stiffer and more elastic. Because of this, rutting is more critical in the hotter summer months and under slower moving traffic [24]. At any combination of time and temperature, viscoelastic behavior, within the linear range, must be characterized by at least two properties: the total resistance to deformation and the relative distribution of that resistance between an elastic part and a viscous part. Although there are many methods of characterizing viscoelastic properties, dynamic (oscillatory) testing is one of the best techniques to represent the behavior of this class of materials. In the shear mode, the dynamic modulus ( $|G^*|$ ), for simplicity, denoted as  $G^*$  hereinafter) and phase angle ( $\delta$ ) are measured.  $G^*$  represents the total resistance to deformation under load, while  $\delta$  represents the relative distribution of this total response between an elastic component and a viscous component. The elastic component can be related to energy stored in a sample for every loading cycle, while the viscous component can be related to energy lost per cycle in permanent flow. The relative distribution of these components is a function of the composition of the material, loading time, and temperature [25].

Depending on the applied stress and strain levels, asphalt binders possess both linear and nonlinear viscoelastic behavior. At small loads, binders generally behave as linear viscoelastic material whereas at high loads they exhibit a nonlinear viscoelastic response [6]. The threshold for the linear region depends on the composition of asphalt binder, loading time and temperature. Within the linear range, the strain is proportional to stress at any instant, which is not true in the

case of nonlinear range [26]. A viscoelastic material possesses characteristics of both elastic and viscous materials and, as such, exhibits time dependent strain behavior, which is commonly referred to as creep. Figure 5(b) shows elastic materials, when loaded in creep, will immediately deform to a constant strain. When the load is removed, the material will immediately return to its initial shape. A viscous material, on the other hand, will deform at a constant rate when the load is applied at to, and will continue to deform at that rate until the load is removed, at which point there is no further deflection or recovery. Figure 5(c) illustrates schematic representation of the viscous response. A viscoelastic material, as shown in figure 5(d), has both elastic and viscous components of response. When loaded in creep, there is an immediate deformation, corresponding to the elastic response, followed by a gradual time-dependent deformation. This time-dependent deformation may further be divided into a purely viscous component and a delayed elastic component. Upon removing the load at  $t_1$ , the viscous flow ceases, and none of this deformation is recovered which is called viscous deformation. The delayed elastic deformation is, however, recovered, but not immediately as with purely elastic deformation. Instead, once the load is removed, the delayed elastic deformation is slowly recovered, at a decreasing rate [10].



**Figure 5.** Material response to a step load: (b) Elastic, (c) Viscous, and (d) Viscoelastic[1]

## 2.6. Existing Asphalt Binder Specification System

An asphalt binder property depends on the source of the crude petroleum, refining process and the location where it is going to be used. Thus it needs to be graded. In pavement applications, Asphalt binders are specified by their physical properties and not their chemical properties. The most important physical properties of the binder to the engineer are its rheological characteristics. The rheological properties of an asphalt binder are expressed in both empirical and fundamental properties. Until the 1970s only empirical properties have been used to characterize and specify Asphalt binders throughout the world.

There are three significant specification systems available to specify or grade Asphalt binders.

They are:

1. Penetration grading system
2. Viscosity grading system, and
3. SuperPave Performance grading (PG) system

### **2.6.1. Penetration Grading System**

AASHTO published specifications for penetration-graded asphalt binders in 1931. The penetration –grading system was the first specification to measure binder consistency at an average pavement service temperature of 25°C[27]. The penetration depth is empirically correlated with asphalt binder performance. In general, lower penetration grades were recommended for use in warmer climates and for heavier traffic and the higher penetration grades for cold climates and light traffic. ASTM D946 standard Specification for Penetration-Graded asphalt binder includes five penetration grades ranging from a hard asphalt graded at “40-50” to a soft asphalt binder graded “200-300” (called “hand”) are used for warm climates[21; 28; 29]. The system is still used by some highway agencies because it is simple and gives fast results concerning the consistency of the binder.

### **2.6.2. Viscosity Grading System**

In the 1960s, the FHWA, ASTM, AASHTO, industry and a number of state highway agencies wanted asphalts to be graded by viscosity at 60°C (140°F). The main reason for this shift was to replace an empirical measure with a more fundamental material property and to measure a property at a temperature which approximates the average pavement surface temperature on a hot summer day. ASTM D3381 Standard Specification for Viscosity-Graded Asphalt Cement and AASHTO M 226 established specification criteria using absolute viscosity at 140°F (60°C) as the principal physical property for grading. In addition, kinematic viscosity at 275°F (135°C) is also usually specified. The purpose of the two criteria was to prescribe limiting values of consistency at two important temperatures. The specification includes five viscosity grades ranging from a hard asphalt graded at “AC-40” to a soft asphalt cement graded at “AC-2.5”. The standard specification for the Aged Residue (AR) includes five viscosity grades ranging from a

hard asphalt graded at “AR-160” to a soft asphalt cement graded at “AR-10”[21; 29; 30; 27]. The AC grading system had been rheological properties.

### **2.6.3. Superpave Performance Grading (PG) system**

In order to assure positive performance of pavements, highway agencies stipulate the use of specific materials for highway pavement construction. The materials are selected on the results of tests run under set conditions. Many of the tests used in the paving industry are only empirical in nature. Basic tests on the empirical properties of the material have been used over the years and general relationships between test results and pavement performance have been developed. This system has worked relatively well in the past, but as we move into the future new approaches are needed to assure good performance of the highway system [10].

In 1987 the United States Department of Transportation implemented SHRP. One of the outcomes of the program is the SuperPave™ Performance Grade binder specification. The PG binder specification differs from the penetration and viscosity grading systems in that the tests used to measure physical properties that can be directly related to field performance by engineering principles. This implies that test measurements should be made at temperatures and loading rates consistent with conditions existing in the pavement. The performance behavior of asphalt binder was defined with viscoelastic properties measured with Dynamic Shear Rheometer (DSR) [31].

Rational tests which measure fundamental properties are needed in obtaining the rheological behavior of binder, which would serve as the basis of an effective performance-based binder specification. Basic rheological properties of asphalt binders include the following:

$G'$  – the storage modulus (elasticity) of the asphalt binder

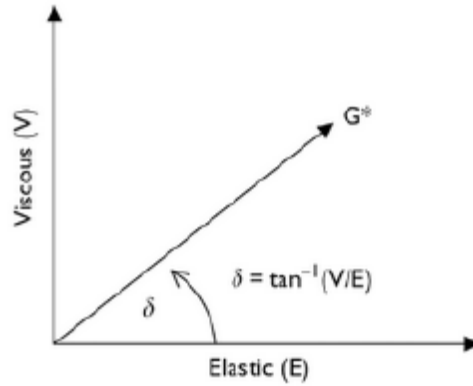
$G''$  – the loss modulus (viscous loss) of the asphalt binder

$G^*$  - the complex modulus which is the amount of energy to deform the asphalt binder

$\delta$  – the phase angle which is the measure of the distribution between the elastic and viscous component

The storage modulus,  $G'$ , represents the in-phase component of the complex modulus, while the loss modulus,  $G''$ , represents the out-of-phase component of the complex modulus. These terms are sometimes misinterpreted as the elastic and viscous moduli; in reality, the elastic component of the response only represents part of the storage modulus and the viscous response only part of the loss modulus. In addition to the elastic and viscous response, most real viscoelastic materials exhibit a significant amount of delayed elastic response that is time-dependent but completely recoverable. In interpreting the storage and loss moduli, it should be kept in mind that both these parameters reflect a portion of the delayed elastic response. Therefore, they cannot be strictly interpreted as elastic and viscous moduli and are properly referred to as the storage and loss moduli [10].

A completely elastic material will not show any difference between the shear stress and shear strain. A completely viscous material would have a phase difference or angle of  $90^\circ$  on the sinusoidal curve. Since asphalt binders are viscoelastic, the phase angle between the shear stress and shear strain is between  $0^\circ$  and  $90^\circ$  (Figure 6). Small phase angles are determined at low temperatures and high frequencies while phase angles closer to  $90^\circ$  are found at high temperatures and low frequencies [16].



**Figure 6.** *Viscoelastic relationship [16]*

These material properties are used in the SHRP's binder specification to evaluate the binder's resistance to tenderness, rutting, fatigue cracking, and thermal cracking. In obtaining these rheological properties, sinusoidal shear strains  $\gamma$  are applied to the binder samples at a frequency of 10 rad/sec. At low testing temperatures (below 34°C), the strains kept constant at 1% and increased to 6% at higher test temperatures (above 52°C). Keeping the strain constant throughout a given test allows the sample to remain in the linear viscoelastic range. Although no material is perfectly linear under all conditions, linear viscoelastic characterization has been found in the past to best represent the rheological behavior of asphalt binders [27].

Based on this, the high temperature criteria  $G^*/\sin \delta$  (1.00 kPa for unaged and 2.2 kPa for RTFO-aged binder) regardless of the location of the pavement, but the test temperature where this criteria must be met is derived from the actual pavement temperature [32]. These tests require equipment that have been developed or modified under the SHRP program. It is called a binder specification because it is intended for both modified and unmodified asphalt binders. A unique feature of the PG binder specification is that, instead of performing a test at a constant temperature and obtaining a varying test value, the specified test value is constant and the test temperature at which the value must be achieved is varied.

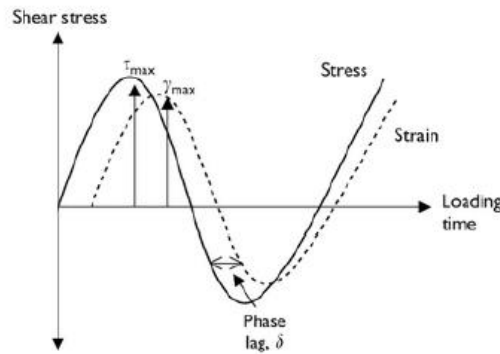


A superPave grade designation contains two parameters and takes the form PG X-Y. The high temperature parameter, X, is the highest temperature at which satisfactory resistance to pavement rutting is expected. The low temperature parameter, Y, is the lowest temperature at which the binder is expected to resist the thermally induced cracking [32]. For example, a PG 64-22 is specified for an average 7-day maximum design temperature of 64°C and a minimum pavement design temperature of -22°C. The designation means that the asphalt binder was classified under the PG system, meets a seven-day average high temperature requirement of 64°C and meets a low temperature requirement of -22°C. The major feature of the PG binder specification is its reliance on testing asphalt binders in conditions that simulate the three critical periods during an asphalt pavement's life. Tests performed on the original asphalt binder represent its transportation, storage, and handling. The second period represents the asphalt binder aging during mixture production and pavement construction, and is simulated in the PG binder specification by aging the asphalt binder in a Rolling Thin Film Oven (RTFO). The final period occurs as the asphalt binder ages over a long time as part of the pavement. This period is simulated in the PG binder specification by the Pressure Aging Vessel (PAV). This procedure exposes asphalt binder samples to heat and pressure conditions that simulate years of in-service aging in the pavement [16].

## **2.7. Dynamic Shear Rheometer (DSR)**

The DSR is used to characterize the viscoelastic behavior of asphalt binder, and evaluate its rutting and cracking potential. The instrument can apply a precise oscillatory, steady, or step shearing strain to the test sample. The parallel plate configuration is used in the test where the size of the plate (i.e. 8 mm or 25 mm diameter) varies depending on the test temperature [27]. The basic principle used for DSR testing is that asphalt behaves like an elastic solid at low

temperatures, and as a viscous fluid at high temperatures. These behaviors can be captured by measuring the complex modulus ( $G^*$ ) and phase angle ( $\delta$ ) of an asphalt binder under a specific temperature and frequency of loading. These parameters are measured by applying a torque on the asphalt binder between a fixed and oscillating plate, and measuring the resulting strain [33].



**Figure 7.** Sinusoidal loading of an asphalt binder by a DSR [16]

Three main tests that are conducted on DSR: Amplitude Sweep Test (AST), Frequency Sweep Test (FST) and Multiple Stress Creep and Recovery (MSCR) Test.

### 2.7.1. Amplitude Sweep Test (AST)

An Amplitude (strain) sweep is an oscillatory test with variable amplitude and constant frequency values. These tests are mostly carried out for the sole purpose of determining the limit of the Linear Viscoelastic (LVE) range [34]. In a strain sweep test, the dynamic modulus values of the specimen are captured over a range of strain amplitudes. In order to fix the linear viscoelastic limit for this material, the strain values corresponding to 95% of the initial modulus was considered. Short term aged (RTFO) samples will be subjected to the strain sweep test to establish the linear viscoelastic limit [35].

At amplitude higher than  $\gamma_L$  (limiting value) the limit of the LVE range is exceeded. The structure of the sample has already been irreversibly changed or even completely destroyed. The

limiting values  $\gamma_L$  for the permissible maximum strain (deformation) are empirically found and proved in practice in many repeated tests. However, these values should only be used as a rough guide as they are not valid in all cases. Therefore, an amplitude sweep should always be carried out first on every unknown sample. This is extremely important because if the limit of the LVE range is exceeded in any subsequent test to be performed, then the laws of hook and newton, which are the basis for the largest part of rheology, no longer apply. The deformation behavior outside the LVE range is referred to as non-linear [34]. At a fixed frequency, the rheological response is measured as a function of the strain amplitude. Once the linear range is established, oscillatory measurements are made in a sweep mode, as a function of frequency at fixed amplitude [36]. Amplitude sweep test is run, where maximum strain is recorded as a function of the maximum amplitude of stress assigned, at an oscillating frequency of 1 Hz [37].

### **2.7.2. Frequency Sweep Test (FST)**

A Frequency sweep is an oscillatory test with variable frequency and constant amplitude values. Sometimes, the term “dynamic oscillation” is used as a synonym for “variable frequency” [34]. The frequency sweep test will be carried out for all unaged and short term aged (RTFO) samples [35]. Maximum strain amplitude is recorded as a function of frequency [37]. Conducting Frequency sweep test in different temperatures and different frequencies helps to understand the changes in the rheological properties under different conditions [38].

The importance of frequency sweeps for people working in industry is that here the time-dependent shear behavior is examined. Short-term behavior is simulated by rapid movements (at high frequencies) and long-term behavior by slow movements (at low frequencies). Before performing a frequency sweep, the limit  $\gamma_L$  of the LVE range must be determined for each new unknown sample, therefore an amplitude sweep must always be carried out first. After this test,

the test conditions for the frequency sweep can be selected to ensure that the test is really carried out in the LVE range [34].

### **2.7.3. Multiple Stress Creep and Recovery (MSCR) Test**

Based on the Superpave binder specification the resistance to permanent deformation at high temperatures was specified with parameter  $G^*/\sin \delta$  (complex shear modulus/sin (phase angle)). Later it has been shown by several authors that this parameter was not always adequate with performance in the road pavement especially in case of modified asphalt binders [31]. As such, many highway agencies have added additional tests to the AASHTO M-320 specification to ensure that a desired modifier is included in the binder. The problem that arises from the use of the resulting Superpave Plus (SHRP+) tests is that in most cases they do not relate to performance, but only indicate the presence of a particular modifier in the binder [39]. During revision of binder Superpave specification for replacing this parameter, the Multiple Stress Creep Recovery (MSCR) test has been developed by D'Angelo et al. [31]. The MSCR test was developed as a replacement for the existing AASHTO M-320 high temperature binder test. This test provides a more accurate measure of rutting resistance by taking into account properties of modified and unmodified binders. The test is conducted with the DSR equipment on the RTFO-aged binders by applying 100 Pa followed by 3200 Pa shear stress levels. At each stress level 10 cycles will be applied by applying a load for 1 second and let it recover for 9 second unloading. From the test two parameters will be obtained non-recoverable creep compliance ( $J_{nr}$ ) and percent recovery (R).  $J_{nr}$  is the measure of permanent deformation and % R is the measure of recoverable strain at the end of 10<sup>th</sup> cycle [40].

Benhood et al. (2016) aimed to investigate the merits of implementing the MSCR test and specification as a replacement for the conventional high temperature testing in the PG system. A

statistical analysis was conducted on dataset from Indian Department of Transportation (INDOT) to see how MSCR and PG procedures differ in grading different binder used in the state. In addition, an experimental study was conducted using seventeen different modified and unmodified binders. The results confirmed that the MSCR test is a suitable replacement for the current PG high temperature test since it provides a better tool to rank modified asphalt binders as well as unmodified ones. That is, creep compliance from the MSCR test more fundamentally represents binder behavior at high temperatures compared to the PG rutting parameter.

Hafeez et al. (2013) assessed the performance (load and temperature) in linear viscoelastic range of modified and neat asphalt binders commonly used in Pakistan. Seven different asphalt binders were tested for temperatures ranging from 20 to 70 degree centigrade and stress levels ranging from 0.025 to 25.6 kPa. The study revealed that the asphalt binders behave in a linear viscoelastic range up to 3.2 kPa. Non-recoverable creep compliance is the governing factor in the selection of an asphalt binder having sufficient elastic response at a particular stress level and temperature.

Mohammed (2014) investigated the relevance of the behavior of asphalt mixtures with varying composition to rectify them with a particular consideration of changes to the mixtures. The experimental programme carried out four tests: Multiple Stress Creep Recovery Test (MSCRT) for base binder and PMB followed by three mixture tests – Wheel Tracking Test (WTT), Indirect Tensile Stiffness Modulus (ITSMT) and Repeated Load Axial Test (RLAT) respectively. The mixtures were manufactured for conventional and three SBS-modified binders (3%, 5% and 7% by mass of binder with grade 40/60) with the same aggregate particles. The MSCRT data illustrated that the PMBs, particularly 5% and 7% are reasonably influential, which can improve

the elasticity of binders associated with non-recoverable ( $J_{nr}$ ) and percentage recovery (%R) parameters to the extent of being able to improve rutting performance.

## 2.9. Viscoelastic Rheological Models

The DSR is a very powerful tool to characterize the LVE rheological properties of asphalt binders. However, the DSR machine also has its limitation where it can only be conducted at a limited range of temperatures and frequencies in accordance with several problems encountered during experimental work and also from the machine itself. Thus the introduction of modeling work is seen to be very useful in order to predict the behavior of asphalt binder that cannot be reached by the experimental campaign [41].

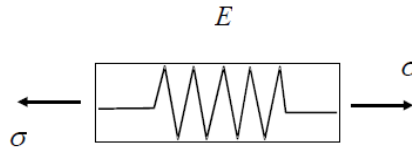
Asphalt binders exhibit both features of elastic solids and viscous fluids and therefore exhibit viscoelastic properties. The main feature of the elastic behavior is to fully store the energy during loading (i.e., creep) and completely dissipate it during unloading (i.e., creep recovery) while the delayed behavior is captured by viscous components. Viscoelastic models can store and dissipate energy at varying intensities during creep and recovery testing. The time dependent behavior of viscoelastic materials may be described by constitutive equations whose variables are stress, deformation and time. These equations may be expressed by means of rheological models [42]. One can build up a model of linear viscoelasticity by considering combinations of the linear elastic spring and the linear viscous dashpot, in parallel or series. These are known as **rheological models** or **mechanical models** [43]. Viscoelastic materials can possess a wide range of relaxation and retardation spectra. With the right choice of parameters, viscoelastic rheological models can accurately describe the behavior of asphalt binders across a wide range of loading durations, strains, and temperatures and therefore largely simulate the real conditions in the field.

### 2.9.1. The Basic Elements: Spring and Dashpot

All linear viscoelastic models are made up of the linear spring and linear viscous dashpot. The constitutive equation for a material which responds as a linear elastic spring of stiffness  $E$  is (see Fig. 8)

$$\varepsilon = \frac{1}{E}\sigma \quad [1]$$

The response of this material to a creep-recovery test is to undergo an instantaneous elastic strain upon loading, to maintain that strain so long as the load is applied, and then to undergo an instantaneous de-straining upon removal of the load (figure 10(a)) [43].

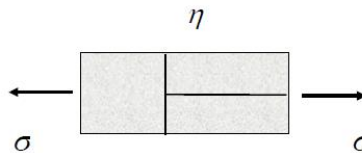


**Figure 8.** *The linear elastic spring [43]*

The dashpot is a piston cylinder arrangement, filled with a viscous fluid, Fig.9 – a strain is achieved by dragging the piston through the fluid. By definition, the dash-pot responds with a strain rate proportional to stress:

$$\dot{\varepsilon} = \frac{1}{\eta}\sigma \quad [2]$$

Where  $\eta$  is the viscosity of the material. This is the typical response of many fluids; the larger the stress, the faster the straining [43].

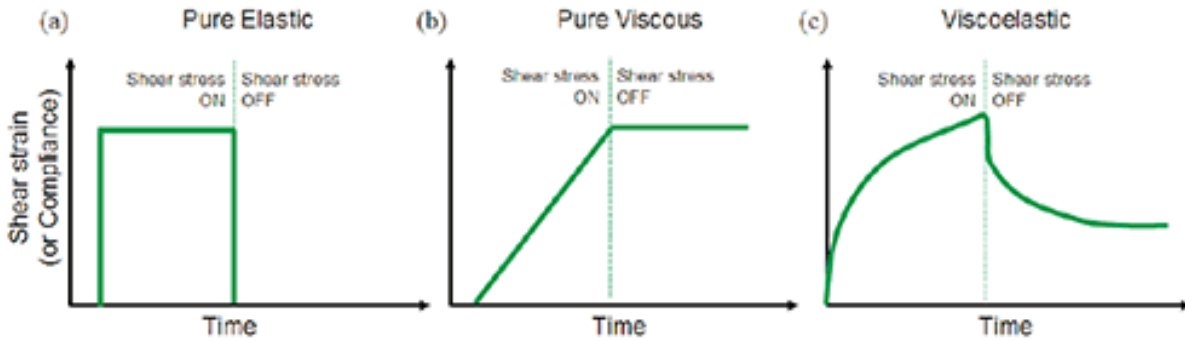


**Figure 9.** *The linear dashpot [43]*

The strain due to a suddenly applied load  $\sigma_o$  may be obtained by integrating the constitutive equation (2). Assuming zero initial strain, one has

$$\varepsilon = \frac{\sigma_o}{\eta} t \quad [3]$$

The strain is seen to increase linearly and without bound so long as the stress is applied (Fig. 10(b)). Note that there is no movement of the dash-pot at the onset of load; it takes time for the strain to build up. When the load is removed, there is no stress to move the piston back through the fluid, so that any strain built up is permanent. The slope of the creep-line is  $\sigma_o / \eta$  [43].



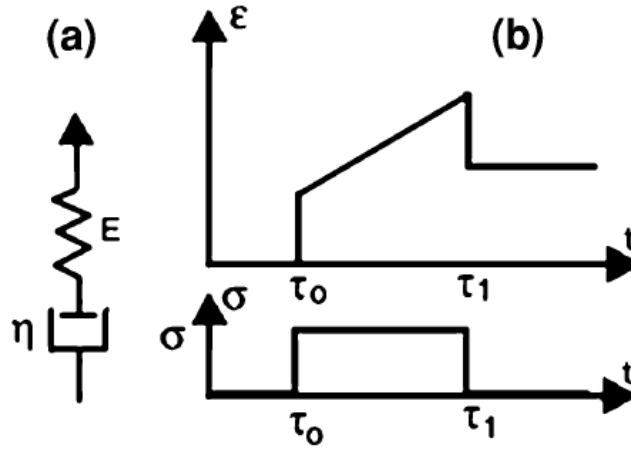
**Figure 10.** (a) *Pure Elastic*, (b) *Pure Viscous*, and (c) *Viscoelastic* [44]

### 2.9.2. The Maxwell Model

The Maxwell model consists of a spring and a dashpot in series to represent the elastic part and the viscous part, respectively. This model is most suitable for cases in which a constant strain is applied and the stress is monitored (stress relaxation). In response to sudden deformation of the model, the spring will be immediately extended, while the dashpot remains initially motionless. But the extended spring will be applying a steady force on the dashpot in an attempt to recoil. This will cause the dashpot to begin to move in the direction of the spring force at a speed governed by the spring force. As the spring begins to recoil with the moving dashpot, the spring force begins to decrease in accordance with the elastic modulus of the spring (spring constant).



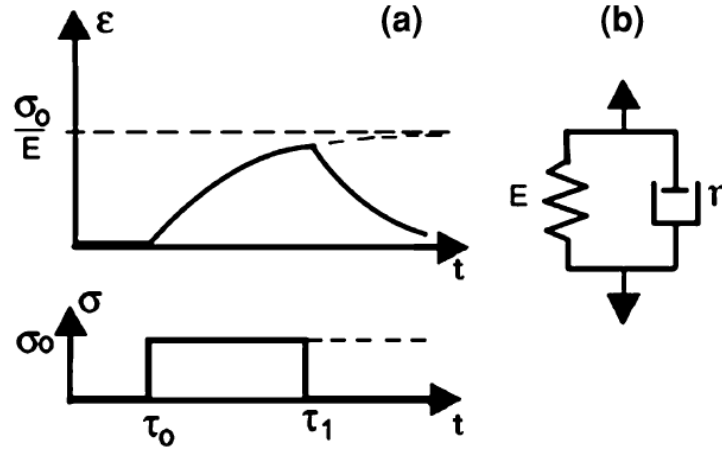
As the spring force continues to decrease, the rate at which the dashpot is moving will also decrease, giving rise to the exponential decay [45].



**Figure 11.** *Maxwell model: (a) rheological model (b) Creep test [46]*

### 2.9.3. The Kelvin/Voigt Model

Simple creep behavior can be described mathematically by the Kelvin model made up of spring and dashpot in parallel. This model is most suitable for cases in which a creep load is applied and the strain is monitored (strain retardation). When a sudden stress is applied to sample material and held constant over time, simple creep behavior is exhibited by the strain beginning to increase rapidly but the rate of increase diminishes over time in the form of an exponential decay. This can be seen in the Kelvin/Voigt model by noticing that the dashpot will control the rate at which the spring can elongate. The initial stress puts maximum force on the dashpot while the spring is fully recoiled (relaxed). Thus the dashpot will begin to move at maximum speed, allowing the spring to extend along with it. In so doing, the spring begins to take on its share of the applied constant stress with the dashpot being relieved of its share of force. As the force on the dashpot diminishes while being taken up by the spring, the speed of the dashpot likewise diminishes. Thus the rate of strain is retarding exponentially (strain retardation), and is called creep [45].



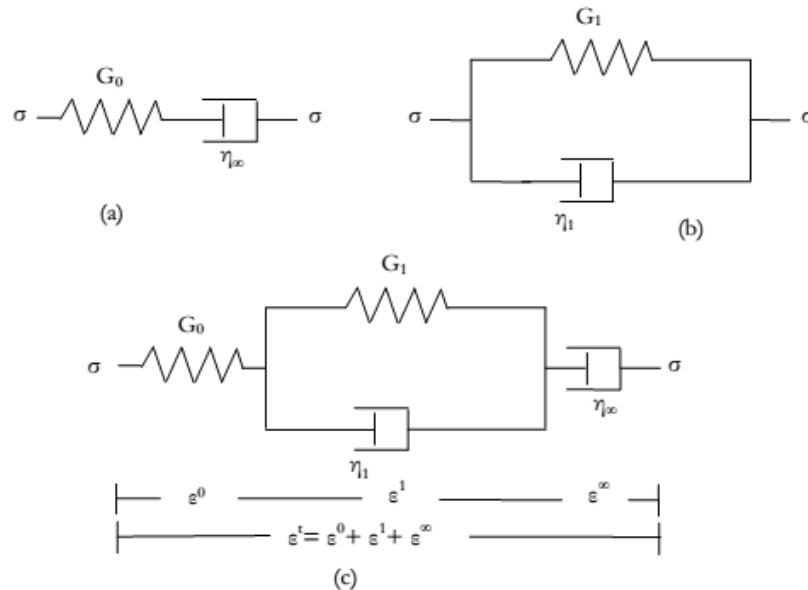
**Figure 12.** Kelvin model: (a) creep test (b) rheological model [46]

Divya et al. (2013) investigated the influence of the type of binder and crumb rubber gradation and dosage rate on the creep and recovery properties of crumb rubber modified bitumen. Two type of binders, air blown and blended, were used with two gradations of crumb rubber, fine and coarse, at three dosage rates, 8, 10, and 12. All samples were subjected to creep and recovery tests by using DSR at five different temperatures of 46, 52, 58, 64, and 70°C. A generalized Kelvin/Voigt model was used to model the creep and recovery response of the material for all the temperatures tested.

Grabowski et al. (2002) dealt with the problem of improving the rheological properties of bitumen through modification with polymers. Two domestic road bitumen, D70 and D200 were used in the laboratory experiments. These bitumens have been modified with three types of elastomers: SBS, SIS, and SBR. The amount of the elastomer added to the bitumen was 4%, 6% and 8%. The samples were investigated in the basic rheological tests: creep and elastic recovery. For the approximation of the non-linear experimental curves obtained, the simple linear viscoelastic models of Kelvin, Lethersich, Burgers and a three parameter standard model have been utilized. A very good approximation of the experiment results of elastic recovery and creep was obtained by making use of a generalized Kelvin model.

### 2.9.4. The Four Element Burgers' Model

Creep-recovery test is a suitable technique to analyze the viscoelastic properties of different materials such as asphalt binder. One of the most common rheological model that describes the total deformation of a viscoelastic system due to constant loading (creep) and unloading (recovery) is the four parameter Burgers model[47].The Burgers' model is widely used for modeling the response of bituminous materials. The model is obtained by placing a single Maxwell element in series with a Kelvin-Voigt element (Figure 13). Its wide application for modelling bituminous materials comes from a number of reasons [1]. One of the main reasons is that the four parameters of Burger's model represent elastic, viscoelastic, and viscous flow properties of asphalt materials which can be determined using creep-recovery data [48]. These three response phenomena are observed in bituminous materials. Like most spring-dashpot combination models, the model is suitable and computationally efficient in numerical applications [1].



**Figure 13.** (a) Maxwell model, (b) Kelvin/Voigt model, and (c) Burgers' model [1]

## **2.10. Related Studies on Fitting Creep Recovery Data into Burger's Model**

Liu and You (2009) proposed a method for determining Burger's model parameters using creep recovery data. The paper derived the constitutive equations of Burger's model under creep recovery test condition, and proposes a procedure to fit creep-recovery data with the Burger's model. Creep-recovery data of the asphalt binder PG64-28 and its two mastics (mastic-100 and mastic-200) was processed with the procedure and the ratios of the viscous flow to the elasticity were calculated with the fitted Burger's model parameters. Then, with the calculated ratios, the viscoelastic behaviors of the asphalt binder and mastics were analyzed. It was found that, Burger's model can be utilized to fit creep-recovery data of both the binder and mastics.

Cui et al. (2015) have studied the rheological properties of base bitumen, SBS modified asphalt and SBS mortar using Dynamic Shear Rheometer (DSR). Creep and relaxation test was used to express rheological behavior combining Second-order Burgers model. A large number of experimental data show that, the fitted results of Burgers' model have a good corresponding relation with the actual result, especially in the loading period. It was concluded that, the model can express viscoelastic behavior of bitumen accurately.

Zhao et al. (2012) investigated the influence of mineral fibers on the low temperature viscoelasticity of asphalt mixtures. Burgers viscoelastic model was used to investigate the parameter regression for the creep test results at the different temperatures. Model parameter was determined through custom fitting the data of time-displacement by Origin mathematical analysis software. Creep curves from test agreed well with Burgers model and the fitted correlation were all above 99% which fully indicated that Burgers viscoelastic constitutive model provided a favorable way to reflect viscoelasticity of mineral fiber asphalt mixture in low temperature. It was concluded that, good agreement between experimental data and Burgers

model was found. The parameters of Burgers model at different temperatures can also provide data support for the viscoelastic design of asphalt pavements.

Adorjanyi and Fuleki (2012) showed the correlation between penetration index and rheological parameters tested with Dynamic Shear Rheometer (DSR) for paving grade bitumen at 10 Hz and + 20°C. In this paper, test data of Multiple Stress Creep Recovery Test at 60 °C were fitted to Burger's model parameters. The four parameters of Burger's model were illustrated versus ten creep-recovery cycles of MSCR test. Good agreement was observed for paving grade bitumen.

Domingos (2016) utilized the MSCR test to analyze the creep recovery behavior of asphalt binders modified with polyphosphoric acid (AC+PPA, PG 76-xx) and Elvaloy terpolymer combined with PPA (AC+Elvaloy+PPA, PG 76-xx) at high pavement temperatures ranging from 52 to 76°C. A recent test protocol standardized by AASHTO (T350-14) was followed in the experiments, and rheological modeling of the data was made based on a seven-step procedure. Substantial increases in  $R$  and considerable decreases in  $J_{nr}$  were observed in the binder after the addition of PPA or a combination of Elvaloy terpolymer and PPA, which can be translated into higher elastic responses ( $R$  values) and a much lower rutting potential in the field ( $J_{nr}$  values). On the other hand, the parameter  $G_v$  (viscous component of the creep stiffness) obtained from the Burgers model ranked the AC+PPA as the most rut resistant formulation at temperatures no greater than 58°C.

Geber (2014) dealt with the rheological properties of asphalt mastics made with mineral fillers, as well as with the relation of fine grain fillers to bitumen. By testing different fillers, various asphalt mastic mixtures has been created, in which the effects of type, grain size and quantity (volume fraction) of the fillers could be observed at the same time. Rheological tests were performed on the mastics in the linear viscoelastic region (LVE) to describe the behavior of asphalt pavements in summer. For the analysis of the creep-recovery features, the behavior of the

mastics has been described with the four-parameter Burgers model where the parameters were numerically defined. It was concluded that the coarse grains in the mastics increase the elasticity of the mixtures in each case and consequently reduce the deformation developed under the effect of load as well as the amount of deformation remaining from recovery. It has also been proved that with the use of limestone, minor deformations developed, which is attributable to the fact that limestone creates a stronger relation with the binder.

# **CHAPTER 3**

## **3. METHODOLOGY**

### **3.1. Introduction**

The type of research used for this thesis is mixed: basic, applied and experimental. I chose experimental because it involves quantitative methods means the samples of the asphalt binders will be subjected to load and the result will be discussed. The thesis is also conducted to solve problems that arise from time and cost. It is also carried out for the enhancement of knowledge and might not have immediate commercial potential.

This chapter elaborates the materials that are selected for this study and investigation of traditional and rheological properties of asphalt binders. The testing methods are also briefly discussed.

### **3.2. Materials**

Two different asphalts binders were selected, which are:

- 40/50 penetration grade bitumen
- 60/70 penetration grade bitumen

#### **3.2.1. Asphalt binder**

Asphalt binder 40/50 pen and 60/70 pen were used in this research paper. In order to evaluate the empirical properties, four laboratory tests have been performed. These are:

- a. Penetration (AASHTO T 49)
- b. Ductility (AASHTO T 51)
- c. Softening point (ASTMD36-2002)
- d. Flash point (AASHTO T 48)

### **3.3. Asphalt Binder Tests**

As discussed in chapter two the rheological properties of an asphalt binder are expressed in both empirical and fundamental properties. The properties that are empirical will be determined from conventional tests whereas; fundamental properties will be determined from Superpave binder tests.

#### **3.3.1. Conventional Tests**

Conventional tests include many standardized tests that were used mostly before the completion of the Strategic Highway Research Program (SHRP). The tests performed on the prepared samples are penetration, ductility, softening point, and Flash and Fire point. These tests are still performed in many countries so it shall be conducted to see if the selected asphalt binders pass the specified limit.

##### **i. Penetration**

The consistency of an asphalt binder test is measured from penetration test. The instrument that is used for this test is called Penetrometer. To conduct the test, the asphalt binder sample was heated to an appropriate pouring temperature and poured into a test container and allowed to cool in air for 1 hour meanwhile the water bath will be maintained to a temperature of 25°C. After we let the sample to cool in air, it was placed in a water bath for 1 hour and 30 minutes. After the specified conditioning period, a 100 gm weight was attached to the standard test needle and allowed to penetrate the sample vertically at 25°C for 5 Seconds at 3 different locations 1 cm apart from each other. The Penetration value was taken as the average of the three values.

##### **ii. Ductility**

Ductility of asphalt binder is its property to elongate under traffic load without getting cracked in road construction works. Some Asphalt binders having a high degree of ductility have also been



found to be more temperature-susceptible and binder material having insufficient ductility gets cracked when subjected to repeat traffic loads and it provides pavement surface that is pervious. To perform the test, the asphalt binder was melted at a temperature of 75°C to 100°C above the approximate softening point until it becomes thoroughly fluid. After stirring the fluid, it was poured in the mold assembly and placed on a brass plate. After about 30-40 minutes, the plate assembly along with the sample will be placed in a water bath; maintained at a temperature of 27°C for half an hour. Then the excess asphalt binder was trimmed with a hot spatula. The test specimen was then placed in the ductility water bath and conditioned to the desired test temperature. After the side pieces of the briquette were detached, the ductility machine was switched on to pull one end the specimen away from the other at a specified rate of speed. The distance in centimeters to which it elongates before breaking was measured. The average of three samples was taken.

### **iii. Softening Point (Ring and Ball Method)**

The softening point of asphalt binder is the temperature at which the substance attains particular degree of softening. The test is useful in determining the consistency of asphalt binder. To perform the test, the asphalt binder sample was heated until it has become sufficiently fluid to pour. The heated samples were poured into two rings, preheated to approximately the pouring temperature. The samples were then cooled for 30 minutes. After that, the excess material was then cleaned with a warm spatula. The apparatus was assembled with the rings, thermometer, and a steel ball weighing 3.5 gram centered in position. The beaker was then filled with distilled water to a height of 50 mm above the upper surface of the rings. Heat was applied to the beaker. The temperature at which the second plate touches the bottom plate was recorded. The average of the 3 samples were then taken and rounded to the nearest whole degree.

#### **iv. Flash and Fire Point**

Flash point is the temperature to which asphalt binder may be heated without the danger of causing an instantaneous flash in the presence of an open flame and Fire point is the lowest temperature at which a sample will sustain burning for 5 second. The apparatus that was be used is the Cleveland Cup. In this procedure, after the sample was heated between 75 to 100°C, a brass cup was filled partially with asphalt binder and was heated at a given rate. A flame was passed over the surface of this cup periodically and the temperature at which this flame causes an instantaneous flash was reported as the flash point. After Flash point, to determine the fire point heating the sample was continued so that the sample temperature increases at a rate of 5 to 6°C. The application of the test flame was continued at 2°C intervals until the sample ignites and continues to burn for at least 5 second. The temperature at that point was recorded as the fire point. The average of the 3 samples was taken and rounded to the nearest whole degree.

#### **3.3.2. SuperPave Binder Tests**

The SuperPave binder PG specifications and mix design are recommended by the Federal Highway Administration (FHWA) for improved and long life highway pavements. Laboratory SuperPave binder tests were conducted using Rolling Thin Film Oven and Dynamic Shear Rheometer.

##### **3.3.2.1. Stress Controlled vs. Strain Controlled Tests**

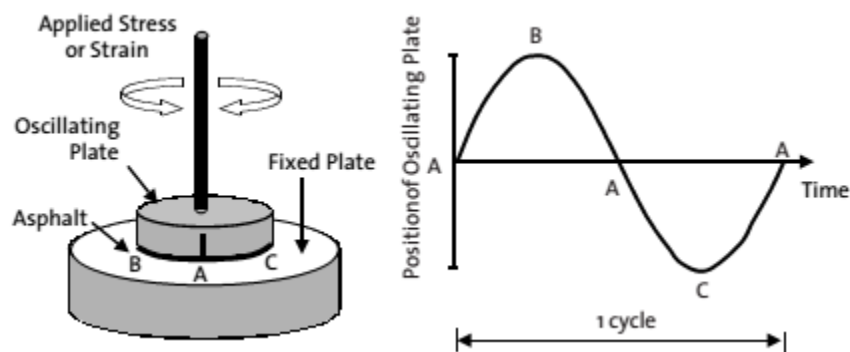
The time or frequency dependent behavior of asphalt binders and mastics can be investigated using oscillatory, creep recovery, and relaxation tests. These testes are carried out using two methods of testing, namely the controlled shear rate (CSR) tests that measure the shear stress and phase angle and the controlled shear stress (CSS) tests that measure the shear strain and phase angle. The rheological parameters resulted from these two tests are essentially dependent on the oscillating frequency ( $\omega$ ) and the loading/unloading time (t) [49].

### 3.3.2.2. Rolling Thin Film Oven (RTFO) Test

The asphalt binder samples were conditioned using the Rolling Thin Film Oven (RTFO). The test simulates the short term aging of the asphalt binder. To perform the test, the sample was first heated at a temperature of 160°C. Then 35 grams of heated asphalt binder was poured into the glass flask, turned to a horizontal position and rotated one full turn to pre-coat the flask. The sample flask will then be allowed to cool in air for 60-80 minutes and placed in a rotating carriage in an RTFO operated at 163°C for 85 minutes.

### 3.3.2.3. Dynamic Shear Rheometer (DSR)

The DSR is a very powerful tool to characterize the LVE rheological properties of asphalt binders. It is set by two plates sandwiching the asphalt binder. Both plates have the same dimensions, but the bottom one is fixed whereas the upper one is mounted on an axis allowing it to rotate. The plate diameter to be used depends upon the temperature. For low to intermediate temperatures an 8-mm diameter parallel plates is used in performing the dynamic testing. 25-mm diameter plates are used for intermediate to high temperatures. The test is conducted by applying a fixed torque on the top spindle to move an oscillating plate from point “A” to point “B” and from point “A” to point “C” (Figure 14). The shear stress can be applied as a sinusoidal varying stress of constant amplitude and fixed frequency.



**Figure 14.** Schematic representation of DSR [50]

### **3.3.3. Testing Procedure**

For this research the DSR machine known as MARVEL BOHLIN INSTRUMENT was used to characterize the asphalt binder properties. The DSR test was performed according to AASHTO T 315-10. In this research paper, all the tests conducted were on the asphalt binder samples that were conditioned using the Rolling Thin Film Oven (RTFO). The asphalt binder samples were then softened to the required consistency and poured into the ring mold to prepare the samples for transfer to the test plates meanwhile the water bath which is used to control the temperature will be set to the selected temperature which allowed preheating the upper and lower plates to adhere with the sample. The 8 mm diameter plate for intermediate temperatures (4°C - 40°C) and 25 mm diameter plate for high temperatures (> 40°C) were used. The molded samples were then transferred to the upper plate. The upper plate together with the sample was lowered to the lower plate until the gap between them equals the test gap setting plus 0.05 mm. The 8 mm diameter plate configuration with 2 mm gap setting and the 25 mm diameter plate with 1 mm gap setting were used for testing. When the lower plates comes in contact with the asphalt binder samples it squeezed out to the edges and were removed by trimming using a heated spatula. When moving the test plates together to the desired test gap, a slight bulge created from the sample. After we made sure that the asphalt binder is in a correct position, the specimen will be allowed to bring to the test temperature. The test will be started only when the test temperature is within  $\pm 0.01^\circ\text{C}$  of the test temperature. The Rheometer is equipped with data acquisition software.

#### **I. Frequency Sweep Test (FST)**

The test was conducted to validate the viscoelastic properties of the asphalt binders evaluated through the MSCR test. In the current study, the FST was conducted at a temperature of 10°C, 21.1°C, 37.8°C, and 54.4°C. 8 mm for 10°C, 21.1°C and 37.8°C and 25 mm for 54.4°C parallel

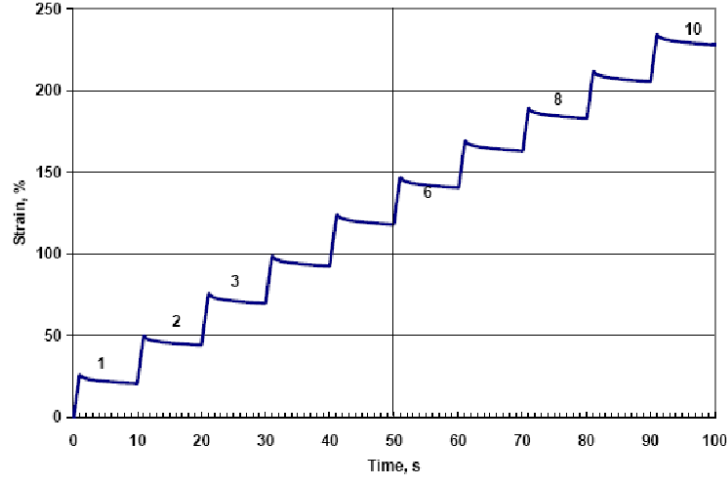
plate was used. The test was performed in a strain controlled mode by setting 1% strain. Test results were observed for a wide range of frequency ranging from 0.1 Hz to 25 Hz.

## **II. Performance Grading (PG)**

The performance grade of the asphalt binders was determined using AASHTOM320 Table 1 (T1) test procedures. 25 mm parallel plate was used. The test were performed starting from a temperature of 52°C and increasing by 6°C until the  $G^*/\sin \delta$  becomes less than 2.2 kPa for RTFO aged asphalt binder. Selection test temperature, in increments of 6 °C, is consistent with the standard PG high temperature. The test is performed to determine the highest temperature at which the MSCR test is conducted.

## **III. Multiple Stress Creep and Recovery (MSCR) Test**

The MSCR test was performed according to AASHTO T 350 at different temperatures. The measurements were conducted using four temperatures (10°C, 21.1°C, 37.8°C and 54.4°C) to compare with FST and three PG high test temperatures (52°C, 58°C and 64°C). The samples were first conditioned by applying stress levels of 100 Pa for 10 cycles followed by another 100 Pa and 3200 Pa for ten cycles each. Each cycle consisted of 1 second shear creep followed by a recovery period of 9 second. For each of the stress levels and temperature used, ten creep and recovery cycles with no rest periods were performed. The shear stress of 100 Pa characterizes the behavior of a binder in the linear viscoelastic region, and the 3200 Pa stress level reflects a binder's behavior in the non-linear viscoelastic region for most modified and unmodified binders. The MSCR test generates two key parameters: non-recoverable creep compliance ( $J_{nr}$ ) and percent recovery (% R). The average  $J_{nr}$  and % R values over the ten cycles are then computed to characterize the overall material properties under constant shear load. Equation 4 to 11 shows how to calculate the  $J_{nr}$  and R values from the creep recovery data.



**Figure 15.** Typical plot of the first 10 cycles of MSCR testing [20]

$$\varepsilon_r(0.1, N) = \frac{(\varepsilon_1 - \varepsilon_{10}) * 100}{\varepsilon_1} \text{ for } N = 1 \text{ to } 10 \quad [4]$$

$$\varepsilon_r(3.2, N) = \frac{(\varepsilon_1 - \varepsilon_{10}) * 100}{\varepsilon_1} \text{ for } N = 11 \text{ to } 20 \quad [5]$$

$$R_{0.1} = \frac{SUM(\varepsilon_r(0.1, N))}{10} \text{ for } N = 1 \text{ to } 10 \quad [6]$$

$$R_{3.2} = \frac{SUM(\varepsilon_r(3.2, N))}{10} \text{ for } N = 11 \text{ to } 20 \quad [7]$$

$$J_{nr}(0.1, N) = \frac{\varepsilon_{10}}{0.1} \quad [8]$$

$$J_{nr}(3.2, N) = \frac{\varepsilon_{10}}{3.2} \quad [9]$$

$$J_{nr0.1} = \frac{SUM(J_{nr}(0.1, N))}{10} \text{ for } N = 1 \text{ to } 10 \quad [10]$$

$$J_{nr3.2} = \frac{SUM(J_{nr}(3.2, N))}{10} \text{ for } N = 11 \text{ to } 20 \quad [11]$$

### 3.4. Viscoelastic Rheological Models

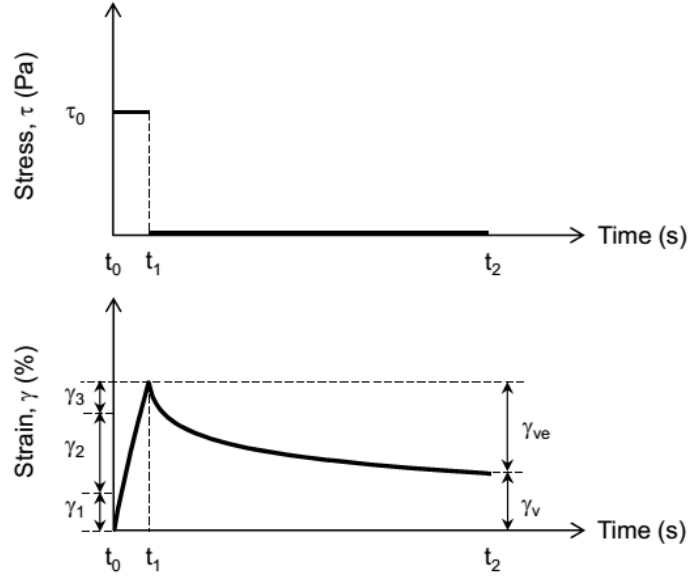
Asphalt binder shows both features of elastic solids and viscous fluids and therefore, behaves as viscoelastic materials. The main characteristics of the elastic behavior is to fully store the

deformation energy during loading and this energy is completely available to act as the driving force for the reformation process during unloading. In viscous flow, however, mechanical energy is continuously and totally dissipated. As a result, viscoelastic models can store and dissipate energy at varying levels during loading/unloading and can be used to describe the time-dependent shear stress/strain response of asphalt binders. The viscoelastic rheological models can precisely describe the behavior of asphalt binders for a wide range of shear strain rates, shear stress levels and temperatures and therefore largely simulate the real conditions in the field [49]. Description of this phenomenon requires laboratory tests at various temperatures and loading durations. Often, the time-temperature superposition principle is applicable for viscoelastic materials such as asphalt binders. At higher temperatures, the time and temperature dependency properties of the asphalt binders can be evaluated under a constant shear stress creep and recovery test.

### **3.5. Rheological Model for the Creep and Recovery Test**

The creep and recovery test is one of the simplest test to measure the viscoelastic properties of a viscoelastic materials. The result from such a test is to know whether the material is a viscoelastic solid or liquid. The test has two parts: creep and recovery. The creep stress was applied instantly and maintained for 1 second and then released to allow recovery for 9 second. Figure 16 illustrates the time-dependent deformation behavior from  $t_0$  to  $t_1$ ; here the asphalt binder at first shows an instantaneous elastic deformation ( $\gamma_1$ ) in response to a sudden applied load, followed by a viscoelastic deformation ( $\gamma_2$ ) and finally transferring a greater load to produce a permanent deformation ( $\gamma_3$ ). After a sudden release of the applied load, the elastic deformation ( $\gamma_e$ ) of the viscoelastic deformation will restore but the viscous deformation ( $\gamma_v$ ) remains as a permanent deformation which is a time-dependent reformation behavior. The

maximum strain ( $\gamma_{\max}$ ) at the end of the cycle will be the sum of the elastic deformation ( $\gamma_e$ ) and viscous deformation ( $\gamma_v$ ) (Equation 12) [51].



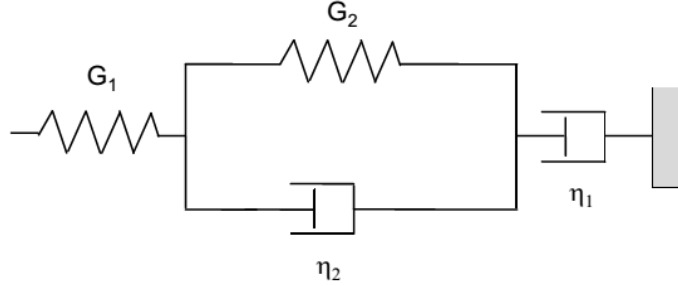
**Figure 16.** *Schematic of creep and recovery test*

$$\gamma_{\max} = \gamma_1 + \gamma_2 + \gamma_3 = \gamma_{ve} + \gamma_v \quad [12]$$

Viscoelastic rheological models are composed of a spring ( $G$ ) and dashpot ( $\eta$ ). The stress-strain-time relation of viscoelastic materials are represented using viscoelastic models. The four-element model, known as the Burgers' model is the one type of model that has been used in many countries to represent the viscoelastic behavior of a material. It is comprised of the Maxwell model ( $G_1, \eta_1$ ) and the Kelvin model ( $G_2, \eta_2$ ). The model is capable of representing initial elastic (instantaneous) deformation,  $G_1$  at the beginning of the test, followed by the delayed viscoelastic deformation,  $G_2$  &  $\eta_2$ , and finally the permanent deformation ( $\eta_1$ ) (Equation 13).  $G_1$  can be thought of as the glass like response,  $G_2$  occurs because of the long chains being entangled like a three dimensional network,  $\eta_2$  occurs because of the polymer melt to the stress applied by the spring and  $\eta_1$  is the terminal response, as the long polymer molecules slide past



each other and disentangled. On the other hand, the creep recovery curve(from  $t_1$  to  $t_2$ ) describes the time-dependent reformation behavior during the rest period(Equation 14).



**Figure 17.** *Four element Burgers' model*

When applying a constant load to the Maxwell model, the first response is that the material responds only elastically because the viscous dashpot initially behaves rigidly. The total deformation of the model remains constant but it redistributes itself between the spring and the dashpot. This results in stress relaxation that occurs exponentially with time, introducing the concept of a relaxation time. When applying a constant load to the Kelvin model, initially the load is carried by the viscous dashpot and then redistributes until, at long times, the load is carried fully by the elastic spring. This introduces the characteristics time which is referred to as the retardation time ( $\lambda$ ) because of the retarded elasticity of the material.

$$\gamma(t) = \gamma_1 + \gamma_2(t) + \gamma_3(t) = (\tau_0/G_1) + (\tau_0/G_2)[1 - \exp(-t/\lambda)] + \tau_0 t / \eta_1 \quad [13]$$

$$\gamma(t) = \gamma_{max} - \gamma_1 - \gamma_2(t) = \gamma_{max} - \left( \tau_0/G_1 \right) - (\tau_0/G_2)[1 - \exp(-t/\lambda)] \quad [14]$$

Where,

$$G_1 = \tau_0/\gamma_1 \text{ and } \lambda = \eta_2/G_2$$

The creep and recovery data collected under constant shear stress over time can be defined in terms of creep compliance function by combining Equations 13 and 14 as:

$$J(t) = \gamma(t)/\tau_0 \quad [15]$$

### 3.6. Relating Time-Frequency Domain Measurements

In general laboratory tests of viscoelastic materials are conducted in the time and frequency domain. Time domain measurements provide material response data from intermediate to long loading durations. Frequency domain tests are used to obtain material response for short loading periods. Material response for short loading periods, less than seconds, cannot be accurately obtained from time domain test data [1].

Different viscoelastic properties can be estimated from different types of mechanical tests, and because these different functions are needed in modeling the behavior of asphalt binder under different loading conditions, it is useful to be able to convert from one viscoelastic function to another. The viscoelastic properties of materials can be obtained through tests conducted either in frequency domain or time domain measurements. Since material behavior is intrinsic, measurements performed in frequency domain can be related to time domain and vice versa. This implies that the creep and recovery data collected in time domain can be related to the corresponding frequency domain measurements. This inter-conversion can be illustrated with rheological models. In this study, the Burgers model parameters are used to determine the storage and loss shear modulus values and in turn utilized to determine the overall viscoelastic properties of asphalt binders (Equations 16 through 19).

To allow a reasonable comparison, the following two points are considered important

- ensure uniform test sampling and test setup procedures
- Ensure stress/strain levels are within the LVE range

$$G'(\omega) = \frac{1}{G_1} + \frac{G_2}{G_2^2 + \omega^2 \eta_2^2} \quad [16]$$

$$G''(\omega) = \frac{1}{\omega\eta_1} + \frac{\omega\eta_2}{G_2^2 + \omega^2\eta_2^2} \quad [17]$$

$$|G^*| = \sqrt{(G')^2 + (G'')^2} \quad [18]$$

$$\delta = \tan^{-1}\left(\frac{G''}{G'}\right) \quad [19]$$

Where,

$G'$  = storage shear modulus

$G''$  = loss shear modulus

$|G^*|$  = complex shear modulus

$\delta$  = phase angle

$\omega$  = angular frequency

### 3.7. Master Curve

The rheological behavior of viscoelastic materials varies with both time (frequency) and temperature. In laboratory only a small range of the viscoelastic response manifests itself within a specified time (frequency) ranges. A broader picture of the time-related response is needed to understand the behavior of a material fully. A solution that arises from the experimental findings is that time (frequency) and temperature of time-dependent processes has equivalent effects on the rheological properties of linear viscoelastic materials. Master curves provide a fundamental rheological understanding of viscoelastic properties of materials and allow an estimation of mechanical properties at wide ranges of temperature and frequency that could be realized in the field, but that are not practical to directly simulate in the laboratory. A master curve could be generated from a series of curves of overlapping data collected at different temperatures. This

procedure is referred to as Time-Temperature Superposition (TTS) principle. The underlying basis for the TTS principle is that there is a direct equivalency between time and temperature for a viscoelastic material. Hence, the loading time can be reduced by performing the laboratory tests at higher temperatures and then transposing (shifting) the resultant data to lower temperatures. The shift factors will be determined using the Williams-Landel-Ferry (WLF) relation (Equation 21). The shear modulus master curve will be developed using the sigmoidal function (Equation 20).

$$\log|G^*| = \delta + \frac{\alpha}{[1 + \exp(\beta - \gamma(\log \omega_r))]} \quad [20]$$

$$\log(a_T) = \frac{-C_1(T - T_{ref})}{C_2 + (T - T_{ref})} \quad [21]$$

$|G^*|$  = dynamic shear Modulus

$\omega_r$  = reduced frequency of loading at a reference temperature

$\delta$  = minimum value of  $G^*$

$\delta + \alpha$  = maximum value of  $G^*$

$\beta, \gamma$  = parameters describing the shape of the sigmoidal function

T = Temperature

$T_{ref}$  = reference temperature

$C_1$  and  $C_2$  are model constants



## **CHAPTER 4**

### **4. RESULT AND DISCUSSION**

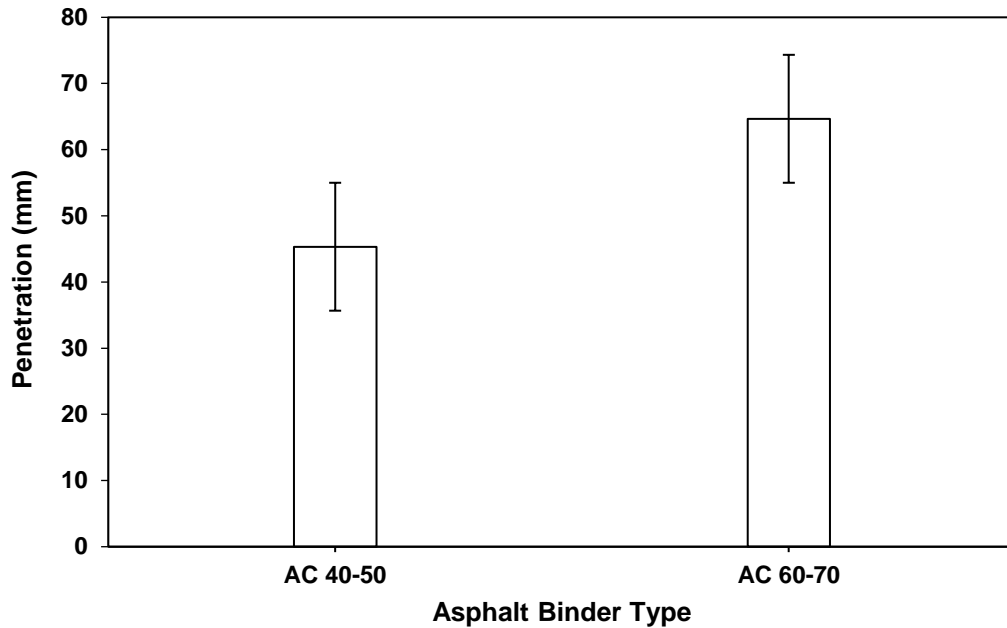
This chapter presents the results of the Conventional and Rheological tests of the two asphalt binders. The test results will be used later on to draw conclusion.

#### **4.1. Conventional properties of Asphalt binder**

Test Results are presented in **Appendix A**.

##### **4.1.1. Penetration**

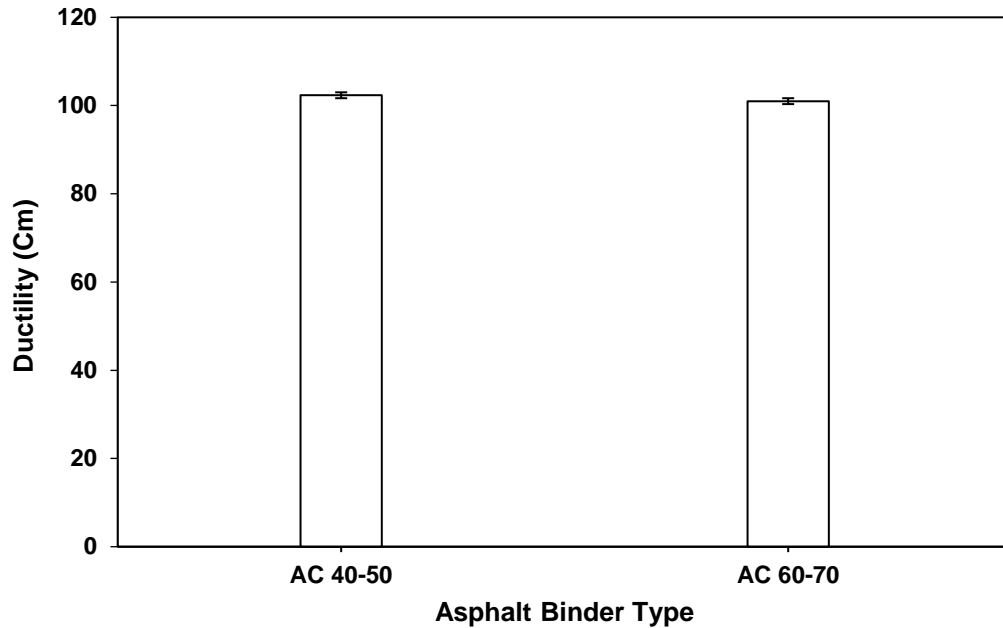
Figure 18 presents the laboratory test results of penetration test for 40/50 pen and 60/70 pen asphalt binder. The test results averaged 45.33 mm for 40/50 pen and 64.67 mm for 60/70 pen and hence the tested binders passed the manufacturer's labeled pen grade. The higher the penetration value, the softer the asphalt binder will become. ANOVA which is one of the statistical analysis was performed to evaluate if the difference between the two means is significant. At  $\alpha = 0.05$  significance level, the null hypothesis which is all the means are equal is rejected. For this research, it is believed that, the choice of the asphalt binders depends on the climatic condition and other factors.



**Figure 18.** *Penetration test result*

#### **4.1.2. Ductility**

The laboratory test results for asphalt binder 40/50 pen and 60/70 pen is presented in Figure 19. The test results averaged 100.33 cm for 40/50 pen and 100 cm for 60/70 pen. The tested binders passed the minimum distance that is specified by the specification which is 100 cm. ANOVA was performed to evaluate if the difference between the two means is significant. At  $\alpha = 0.05$  significance level, the null hypothesis which is all the means are equal is accepted and therefore the difference between the two means is not significant.

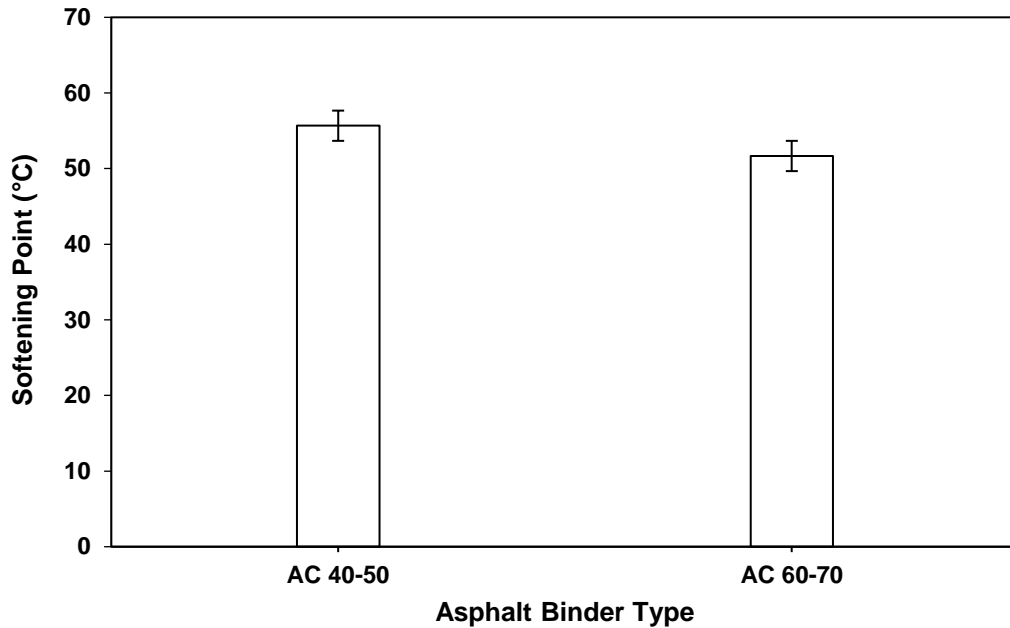


**Figure 19.** *Ductility test result*

#### **4.1.3. Softening Point**

Figure 20 presents the softening point test laboratory test results for 40/50 pen and 60/70 pen asphalt binders. It can be seen from the figure that 60/70 pen resulted in lower softening point. It is observed that as the penetration valued increased the softening point decreased this is because stiff asphalt binders are temperature susceptible it needs high heat to become soft. ANOVA was performed to evaluate if the difference between the two means is significant. At  $\alpha = 0.05$  significance level, the null hypothesis is accepted and the difference between the two means is not significant. From the test results it is observed that 40/50 pen resulted in higher softening point which reflects better rutting resistant at high temperature.

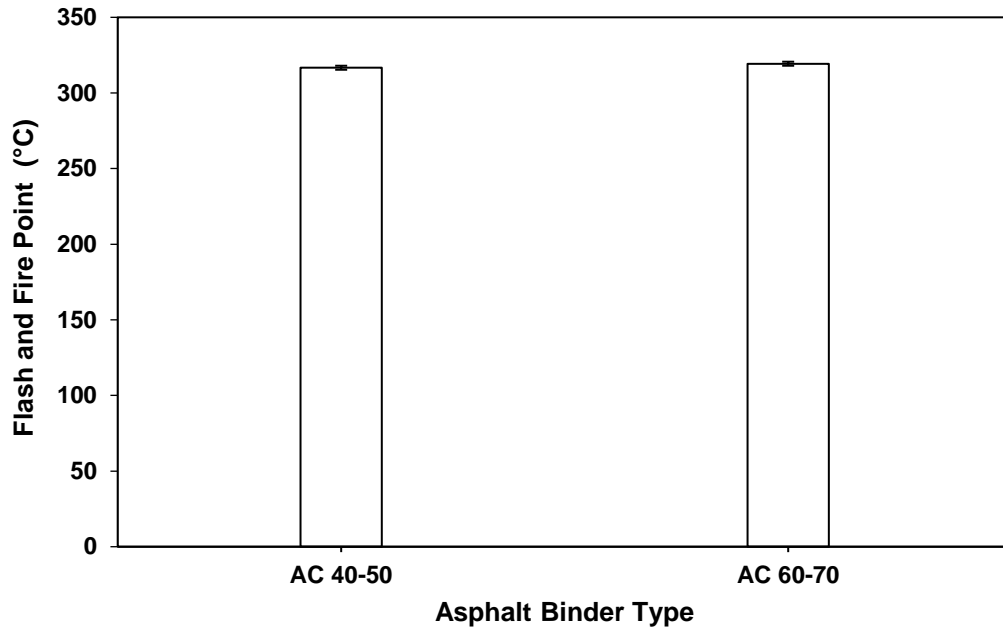




**Figure 20.** *Softening point result*

#### **4.1.4. Flash and Fire Point**

Figure 21 shows the flash and fire point of the tested binders. The laboratory test results showed that 317°C and 319°C was recorded for 40/50 pen and 60/70 pen. From the results it is observed that the asphalt binder can be heated up to 300°C without causing harm. ANOVA was performed to evaluate if the difference between the two means is significant. At  $\alpha = 0.05$  significance level, the null hypothesis is accepted and the difference between the two means is not significant.



**Figure 21.** *Flash and Fire point test result*

## **4.2. Rheological properties of Asphalt binder**

The rheological properties of the asphalt binders included in this study were determined following the AASHTO T315 and AASHTO T350 using a Dynamic Shear Rheometer (DSR) test protocol.

### **4.2.1. SuperPave PG Grading**

The actual PG high temperatures of the tested binders were estimated and are presented in Table 1. These temperatures are based on the  $G^*/\sin\delta$  values obtained from DSR tests of RTFO-aged binders. According to the SuperPave specifications, the  $G^*/\sin\delta$  value for RTFO aged asphalt binder is  $\geq 2.2$  kPa. As it can be seen from the results both binders resulted in PG 64-YY. This means that the binders would possess adequate physical properties to resist rutting at least up to 64°C. Test results are presented in **Appendix B**.

**Table 1.** *Designation of asphalt binders*

Penetration Grade	Temperature (°C)	$G^*/\sin \delta \geq 2.2$ (kPa)	PG Grade
40/50	70	2.97	PG 64-YY
60/70	70	3.33	PG 64-YY

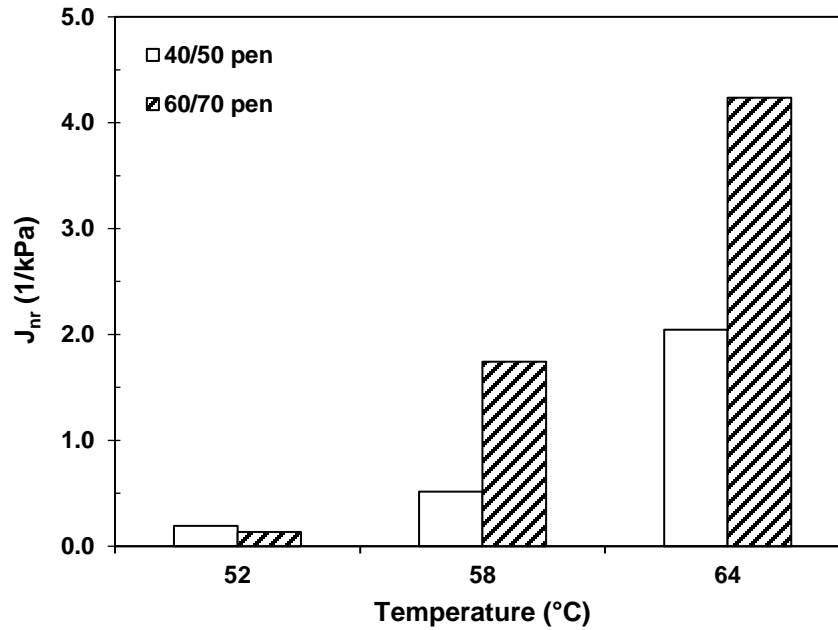
#### **4.2.2. Linear Viscoelastic (LVE) Range**

The basic laws of rheology as well as the viscoelastic models elaborated in chapter 3 are valid only when the tests are conducted in a linear viscoelastic (LVE) range. In the current study, the MSCR test was performed on RTFO-aged binders according to AASHTO T 350 at different temperatures by applying stress levels of 100 Pa followed by 3200 Pa for ten cycles each. The stress level, at which the materials that are tested under LVE range, will not change the structural behavior and therefore the steady-state viscoelastic properties of the material, can be captured. It is proved in many studies such as Benhood et al., 2016 and Hafeez et al., 2013 that, the shear stress of 100 Pa characterizes the behavior of a binder in the LVE range, and the 3200 Pa stress level reflects a binder's behavior in the non-linear viscoelastic range for most modified and unmodified binders. On this research paper, a 100 Pa shear stress was chosen to characterize the behavior of a binder within the LVE range.

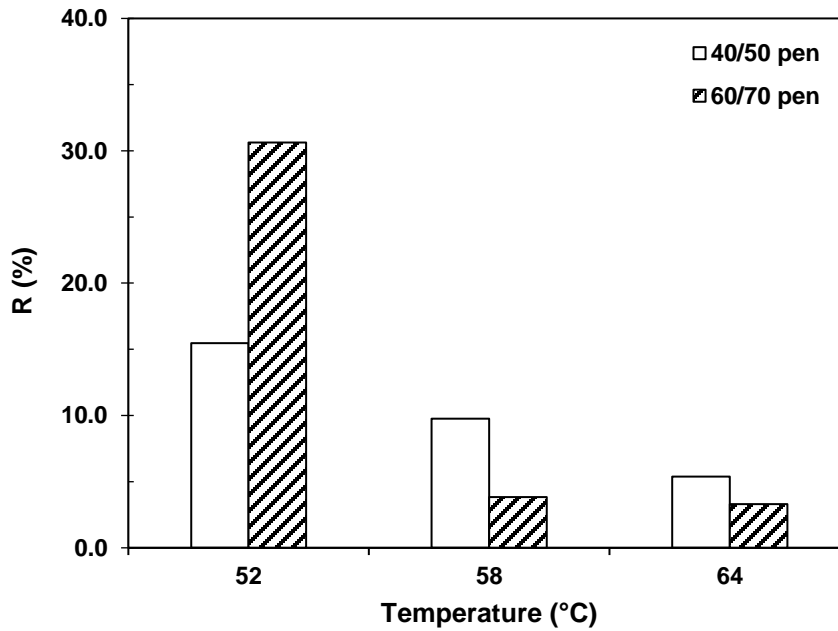
#### **4.2.3. Creep and Recovery Properties**

The non-recoverable creep compliance and percent recovery values at 100 Pa for the studied asphalt binders are shown in Figures 22 and 23 respectively. It is shown in the figures that, the  $J_{nr}$  value is higher and R value is lower for 60-70 pen as compared to the 40/50 pen. The reason for this is, the interaction between the molecules is loose. Overall it is observed that, as the temperature increases, the non-recoverable creep compliance values increase and the percent recovery values decrease. This is because the interaction between the molecules of the asphalt

binder will decrease thereby lose its stiffness and increase its deformation. Test results are presented in **Appendix C**.



**Figure 22.** Non-recoverable creep compliance ( $J_{nr}$ ) at 100 pa

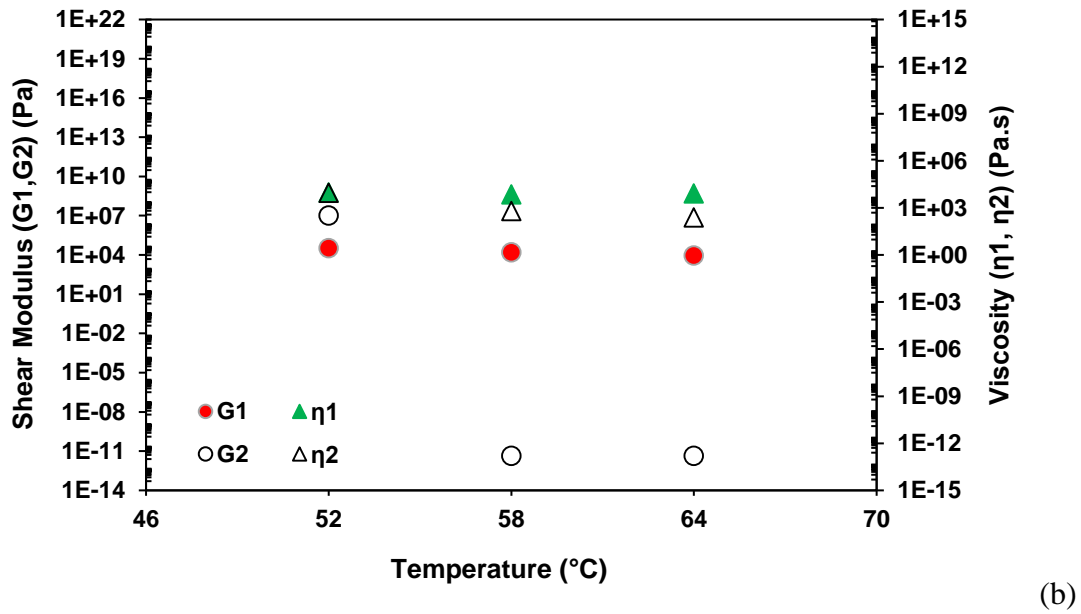
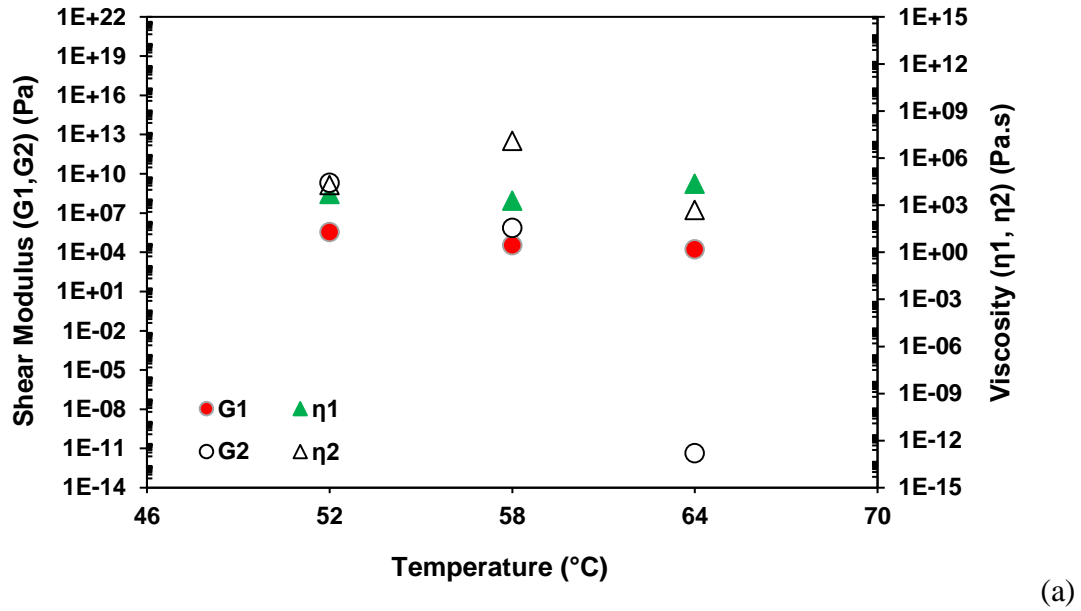


**Figure 23.** Percent Recovery ( $R$ ) at 100 Pa

### 4.3. Fitting Burgers' Model Parameters

To optimize the Burgers' model parameters, the creep recovery data obtained from MSCR test will be used. The incremental formulation was utilized in the optimization process. The procedure is based on minimizing an objective function equal to the sum of the square of errors for estimating shear strain over the ten creep and recovery cycles. The “Solver” function of the Microsoft Excel was used to conduct the nonlinear optimization for simultaneously solving the parameters. The measured strain value and the creep compliance will be used as an input to compute the parameters.

The Burger model parameters for the studied asphalt binders are presented in Figure 24. Four parameters was obtained:  $G_1$ ,  $\eta_1$ ,  $G_2$ , and  $\eta_2$ . Higher values of  $G_1$  &  $G_2$  together with  $\eta_1$  &  $\eta_2$  indicates stiffer and more rigid asphalt binder properties. The temperature dependency of the model parameters indicates the change in retardation spectrum (delayed response) of the materials. From Figure 24 it is observed that, as the temperature increases  $G_1$ ,  $G_2$ , and  $\eta_2$  values decrease and  $\eta_1$  value increases for the reason that at a high temperature asphalt binders will become soft and lose its elasticity (resistance to deformation). Overall it is observed that the change in temperature affects the material property highly. When we compare the  $G_2$  values of the tested asphalt binders, higher value was obtained for 40-50 pen which indicates improved stiffness and higher resistant deformation. These observations are in line with the findings of previous section (4.2.3.). Test results are presented in **Appendix D**.



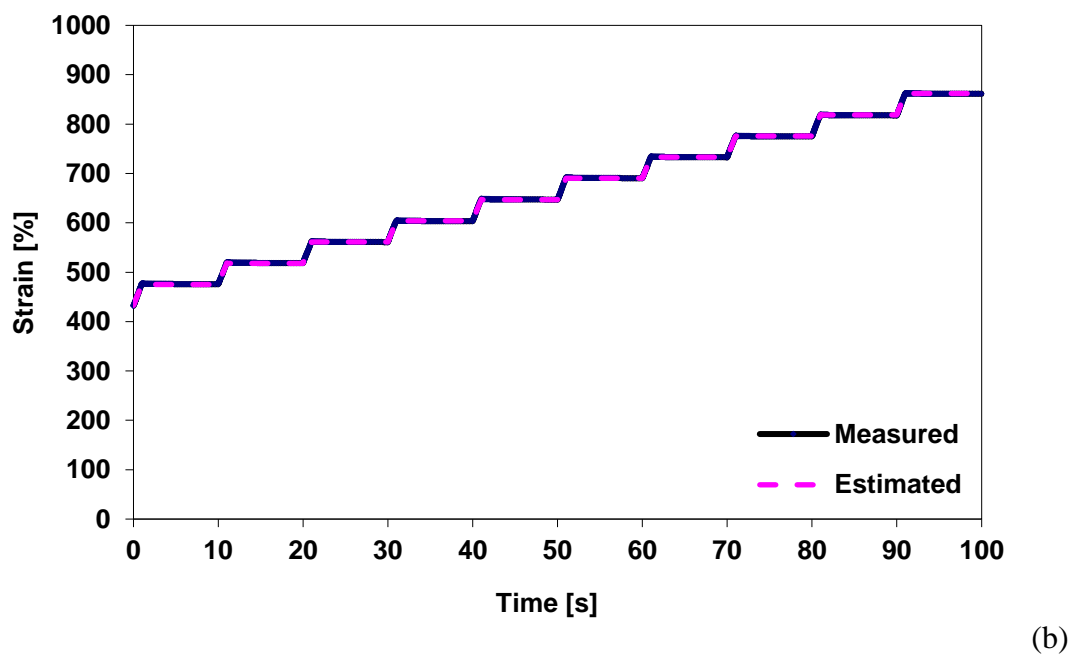
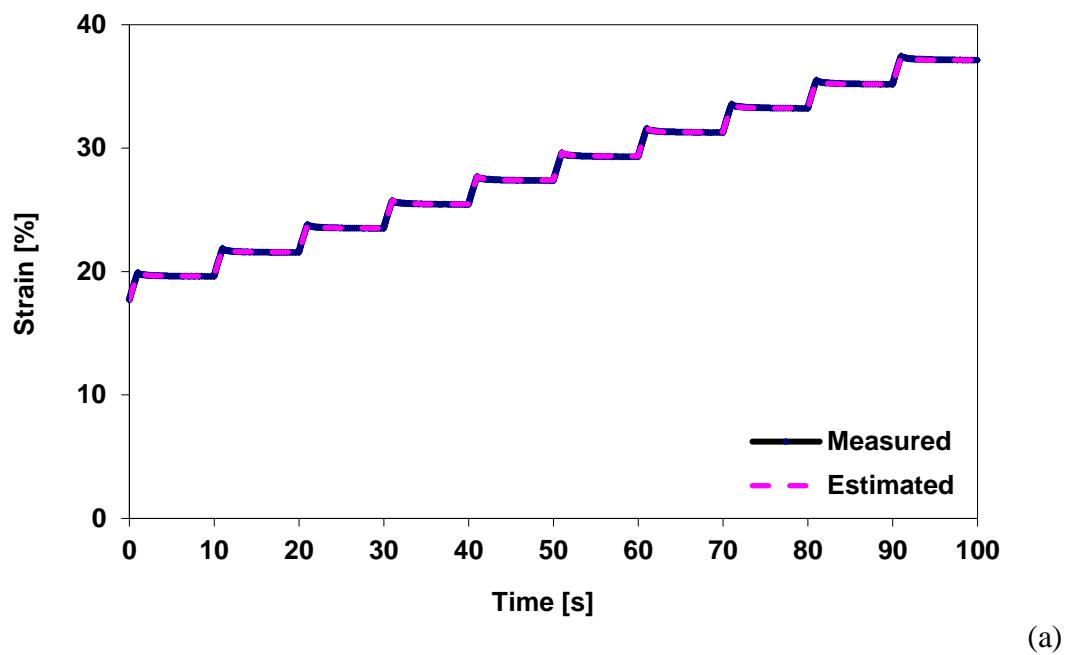
**Figure 24.** Burgers' model parameters at 100 Pa; (a) 40/50 pen and (b) 60/70 pen

#### 4.4. Comparison of Measured and estimated Shear Strain Values

Figure 25 presents the comparison between the measured and estimated shear strain values for 40-50 pen and 60-70 pen at 64°C. The measured shear strain values were obtained from MSCR

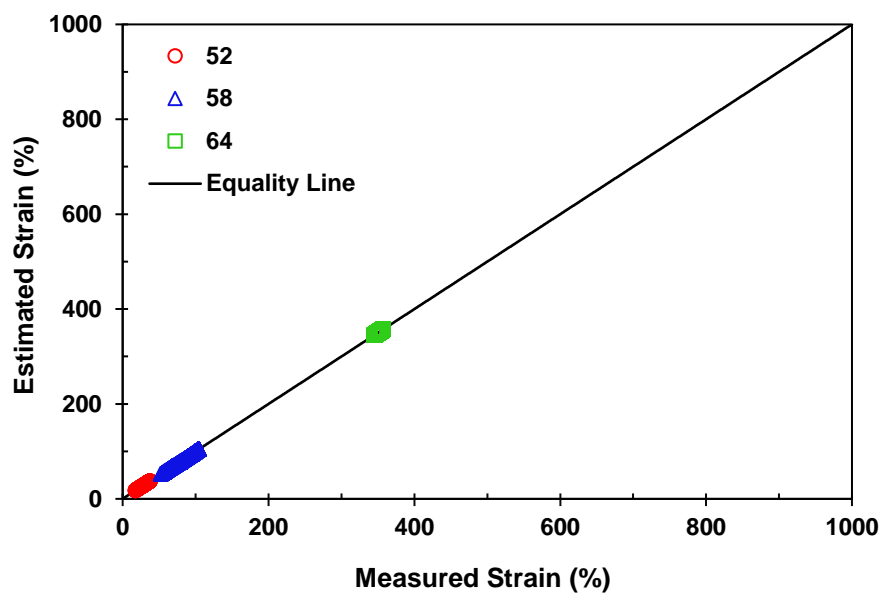
laboratory test results for the 10 creep and recovery cycles whereas the estimated strain values were obtained by utilizing the burger model parameters. From the results it is observed that the estimated strain values satisfactorily matches the measured strain values. This implies that the burger model can be utilized to model the strain values. Higher deformation value was recorded for 60-70 pen. Overall, an increase in time will increase shear strain values for both measured and estimated strain values. Test results are presented in **Appendix E**.

Goodness-of-fit statistics were used to indicate the goodness of fit between the measured strains and estimated strains. The statistical methods include coefficient of determination ( $R^2$ ), standard error of estimate ( $S_e$ ), and relative accuracy ( $S_e/S_y$ ), in which  $S_y$  is the standard deviation. Figure 26 presents the comparison of measured and estimated strains (both creep and recovery) for the asphalt binders included in the study. The goodness-of-fit indicators for estimating creep and recovery strains are presented in Table 3. The coefficient of determination ( $R^2$ ) for the studied asphalt binders are higher than 99 %. The overall strain estimation errors ( $S_e/S_y$ ) for these asphalt binders are not higher than 0.045. Based on the criteria for goodness of fit statistics, excellent estimation of the creep and recovery strains was observed (Table 2). Therefore, the Burgers model parameters were found to be suitable and adequate for describing the creep and recovery viscoelastic properties of the asphalt binders included in the study.

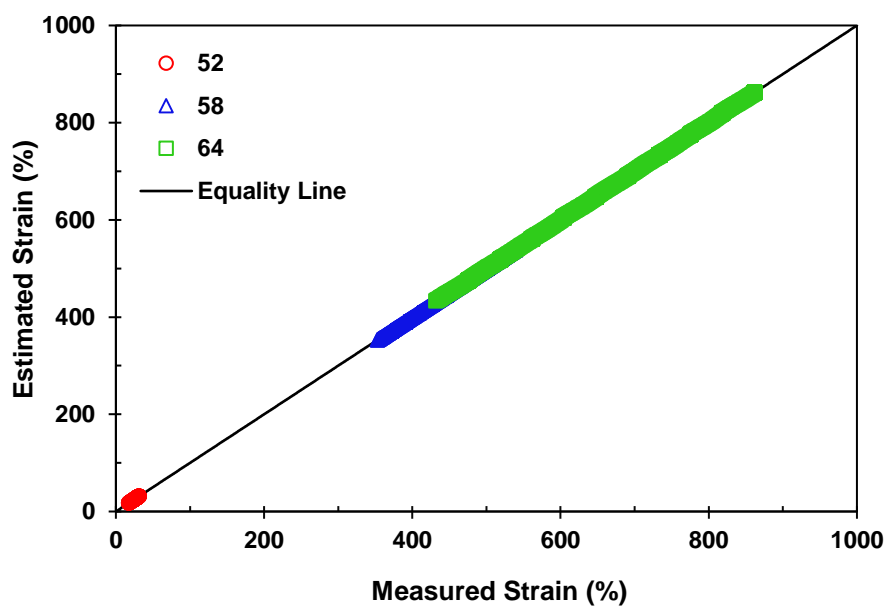


**Figure 25.** Shear strain at 100 Pa and 64°C; (a) 40/50 pen and (b) 60/70 pen





(a)



(b)

**Figure 26.** Comparison of strains at 100 Pa; (a) 40/50 pen and (b) 60/70 pen

**Table 2.** *Criteria for goodness-of-fit statistics*

<b>Criteria</b>	<b>R<sup>2</sup></b>	<b>Se/S<sub>y</sub></b>
Excellent	≥ 0.90	≤ 0.35
Good	0.70 – 0.89	0.36 – 0.55
Fair	0.40 – 0.69	0.56 – 0.75
Poor	0.20 – 0.39	0.76 – 0.89
Very poor	≤ 0.19	≥ 0.90

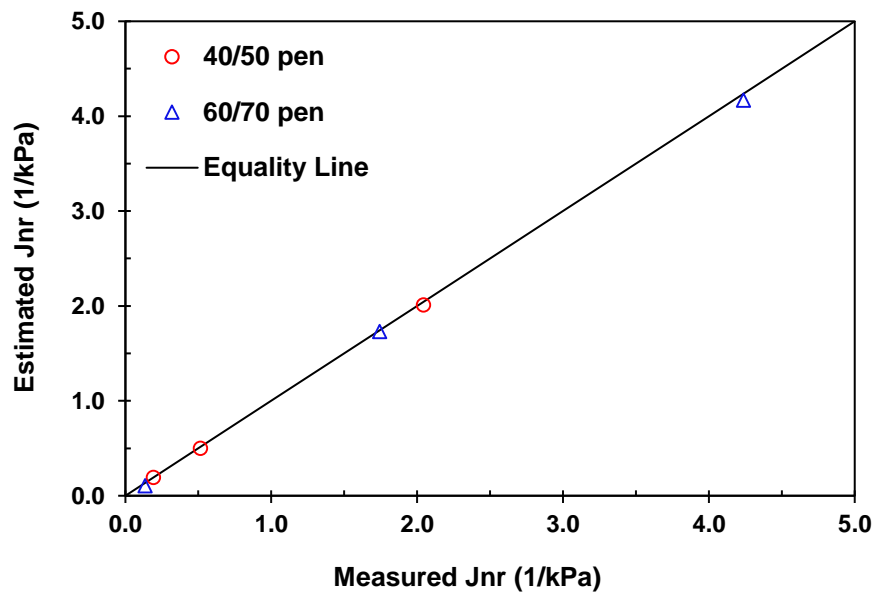
**Table 3.** *Goodness-of-fit statistics result*

<b>Binder ID</b>	<b>Temperature (°C)</b>	<b>R<sup>2</sup> (%)</b>	<b>Se/S<sub>y</sub></b>
40/50 pen	52	99.99	0.004
	58	99.97	0.017
	64	99.76	0.005
60/70 pen	52	99.82	0.042
	58	99.99	0.004
	64	99.99	0.005

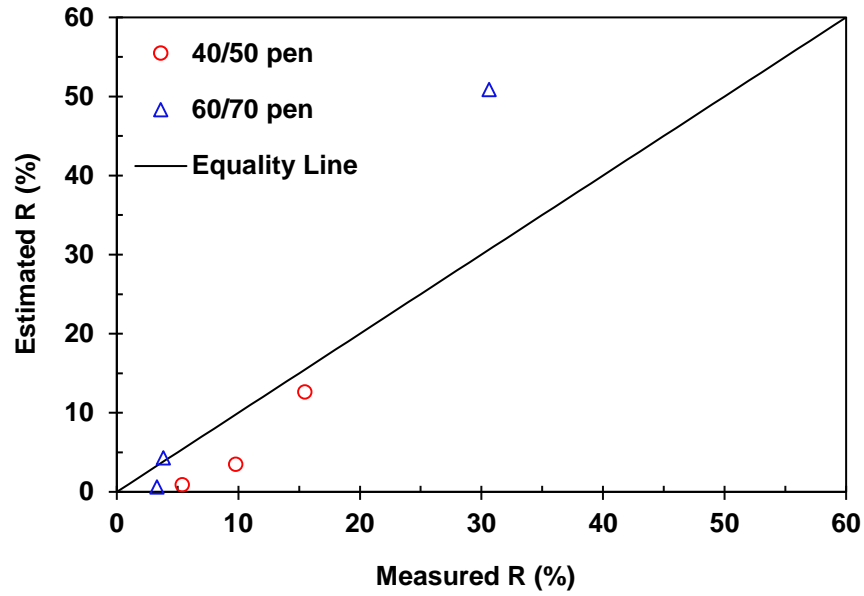
#### **4.5. Comparison of Measured and Estimated Creep and Recovery Properties**

Comparison of the measured and estimated non-recoverable creep compliance ( $J_{nr}$ ) and percent recovery ( $R$ ) values for the 10 creep and recovery cycles are presented in Figure 27 and 28. From Figure 27 it is observed that the estimated  $J_{nr}$  values fits with the measured  $J_{nr}$  values which implies that the Burger model is suitable for characterizing the deformation property of the asphalt binder. Figure 1.28 shows that the estimated  $R$  values doesn't exactly fit with the measured  $R$  value. Goodness-of-statistics which includes coefficient of determination ( $R^2$ ) and relative accuracy ( $Se/S_y$ ) were performed. The coefficient of determination ( $R^2$ ) for the studied asphalt binders are higher than 90%. The overall estimation error ( $Se/S_y$ ) for these asphalt

binders were not higher than 0.36 and 0.01 for  $R$  and  $J_{nr}$ . Based on the criteria for the goodness of statistics, excellent estimation of the  $J_{nr}$  and good estimation of the  $R$  was observed (Table 2). In general, acceptable estimate of the creep and recovery properties of the asphalt binders are obtained. This suggests that the Burger model parameters used to estimate strains are suitable to characterize the creep and recovery viscoelastic properties of the asphalt binders included in the study. Test results are presented in **Appendix F**.



**Figure 27.** Comparison of non-recoverable creep compliance ( $J_{nr}$ ) at 100 Pa



**Figure 28.** Comparison of Percent Recovery ( $R$ ) at 100 Pa

**Table 4.** Goodness-of-fit statistics result

Binder ID	$J_{nr}$ (1/kPa)		$R$ (%)	
	$R^2$ (%)	$S_e/S_y$	$R^2$ (%)	$S_e/S_y$
40/50 pen	99.99	0.008	94.58	0.36
60/70 pen	99.99	0.004	99.98	0.002

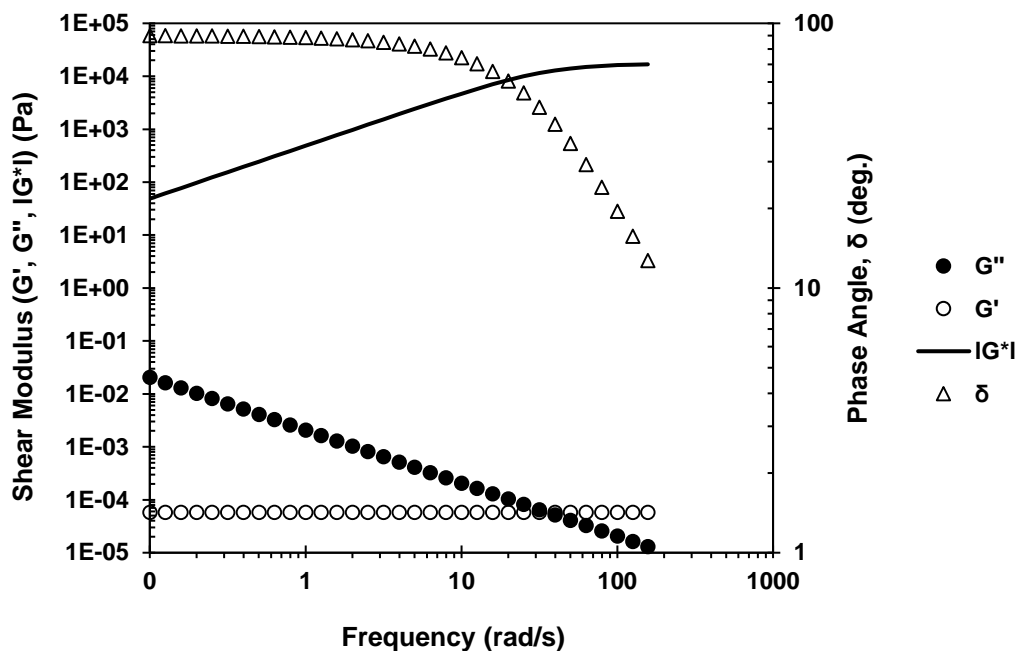
#### 4.6. Viscoelastic Properties

The fitted Burger model parameters are used to determine the viscoelastic properties such as storage modulus, loss modulus, complex shear modulus and the phase angle using equation 16 to 19. At each test temperature four set of viscoelastic properties were be obtained for a reasonably narrow ranges of frequencies (0.016-25 Hz). These predicted values will be used later on to construct master curves at each test temperatures via Time-Temperature superposition principle.

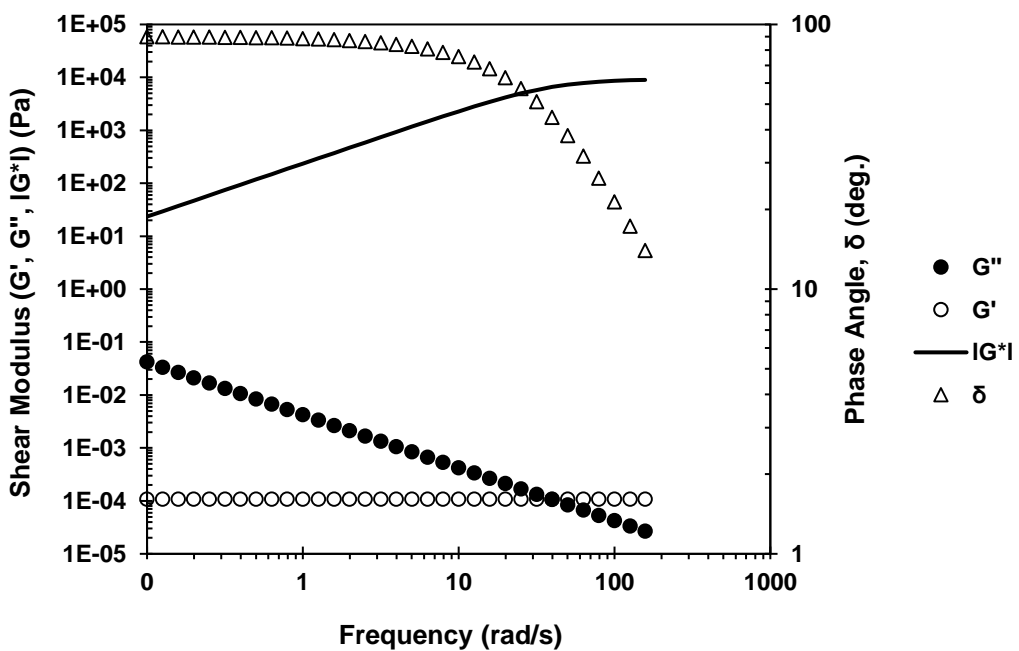
Figure 29 presents the viscoelastic properties of the 40/50 pen and 60/70 pen at a temperature of 64°C for a range of frequencies. Similar plots were also generated for different temperatures. The

frequency ranges in this figure are decomposed into three distinct regions: the initial range at low frequency, transition range at intermediate frequency, and the glassy range at high frequency. At the low frequency range, the tested asphalt binders curves indicated the dominance of the viscous portion ( $G' < G''$ ) and the binder exhibited the behavior of viscoelastic liquid properties. This is also manifested via higher phase angle ( $\delta$ ) values. The  $G''$  curves of 40/50 pen and 60/70 pen decreased significantly as the frequency increases. At intermediate frequency range, a viscous behavior with viscoelastic liquid property was observed for the two asphalt binders. Much of the deformation at the intermediated frequency is because of the delayed elastic response which is a time-dependent deformation but completely recoverable. At high frequency range, the  $G'$  curve reaches a plateau which denotes the glassy-like properties for the asphalt binders included in this study. This glassy-like behavior is accompanied by a decreasing phase angle value.

Observing the behavior of the phase angle as a function of frequency, the general shape is sigmoidal. The phase angle at very low frequencies approaches  $90^\circ$ , and at very high frequencies the phase angle approaches  $0^\circ$ . At the crossover frequency, the phase angle is approximately equal to  $45^\circ$ . At a test temperature of  $64^\circ\text{C}$ , crossover frequency values of 35.61 rad/s and 39.26 rad/s were obtained for the 40/50 pen and 60/70 pen respectively (Figure 29). The lower crossover frequency for the 40/50 pen binder is an indication of faster transition time from viscous to elastic region. Generally, the crossover frequency values for the asphalt binders evaluated in this study ranged from 6.3 to 100 rad/s. Test results for other temperatures are presented in **Appendix G**.



(a)

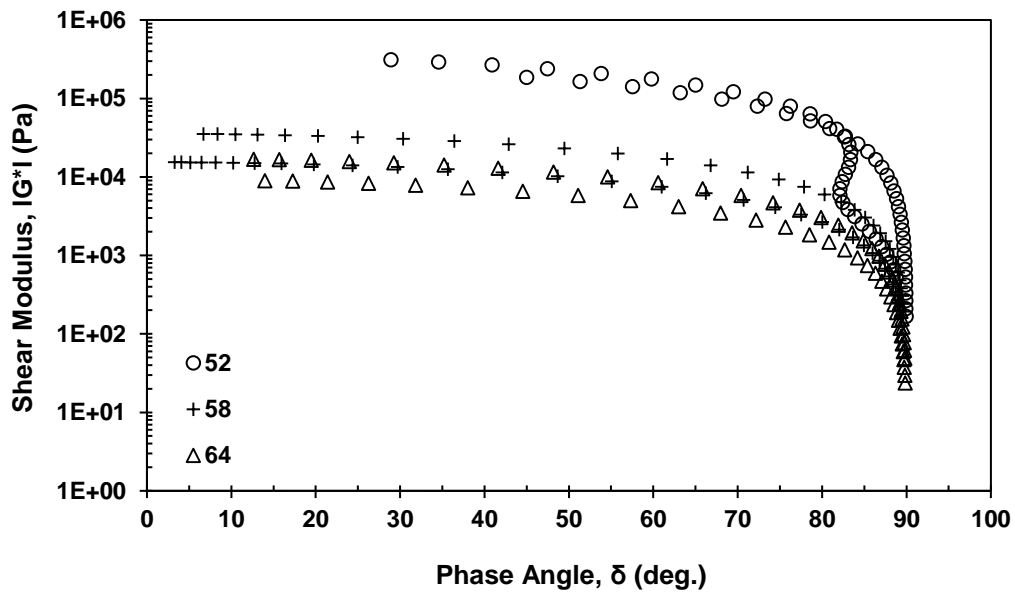


(b)

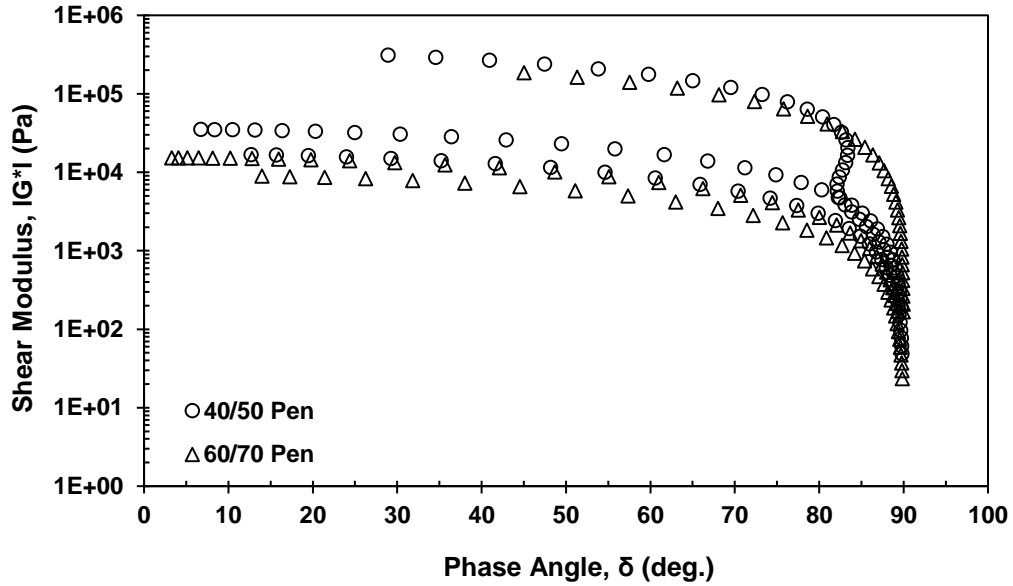
**Figure 29.** Comparison of viscoelastic properties at 100 pa and 64C; (a) 40/50 pen and (b) 60/70 pen

#### 4.7. Black Space Diagram

A Black Space diagram is a graph of the complex modulus,  $G^*$ , versus the phase angle,  $\delta$ , obtained from the burger model. The Frequency and the temperature are therefore eliminated from the plot, which allows all the data to be presented in one plot without the need to perform TTSP manipulations of the raw data. A smooth curve in a Black Space diagram is a useful indicator of time and temperature equivalency, while a disjoint curve indicates the breakdown of TTSP and the presence of modifier. Figure 30 presents Black Space diagram for the studied asphalt binders. In general, it is observed that, the curve is smooth which indicates that the asphalt binder is not modified which is true. Overall, it is shown in the figure that as the test temperature increases the shear modulus value decreases because of the difference in the binder rheological properties.



(a)



(b)

**Figure 30.** Black Space Diagram for 40/50 pen and 60/70 pen; (a) test temperatures and (b) asphalt binder type

#### 4.8. Master Curve using MSCR Test Result

This section describes the viscoelastic properties of the asphalt binders in frequency domain computed using the Burger model parameters. For each test temperature, the dynamic shear modulus values were computed using frequency ranges typically used in oscillatory FST (0.01 to 25 Hz). The shear modulus master curves were used to compare and evaluate the viscoelastic properties of the asphalt binders included in this study. The construction of master curves at several temperatures and frequencies utilizes the TTS principle of viscoelastic materials. By combining the master curve model (Equation 20) and the WLF relation (Equation 21), regression analysis was performed to obtain the relevant parameters for the WLF and the generalized logistic functions.

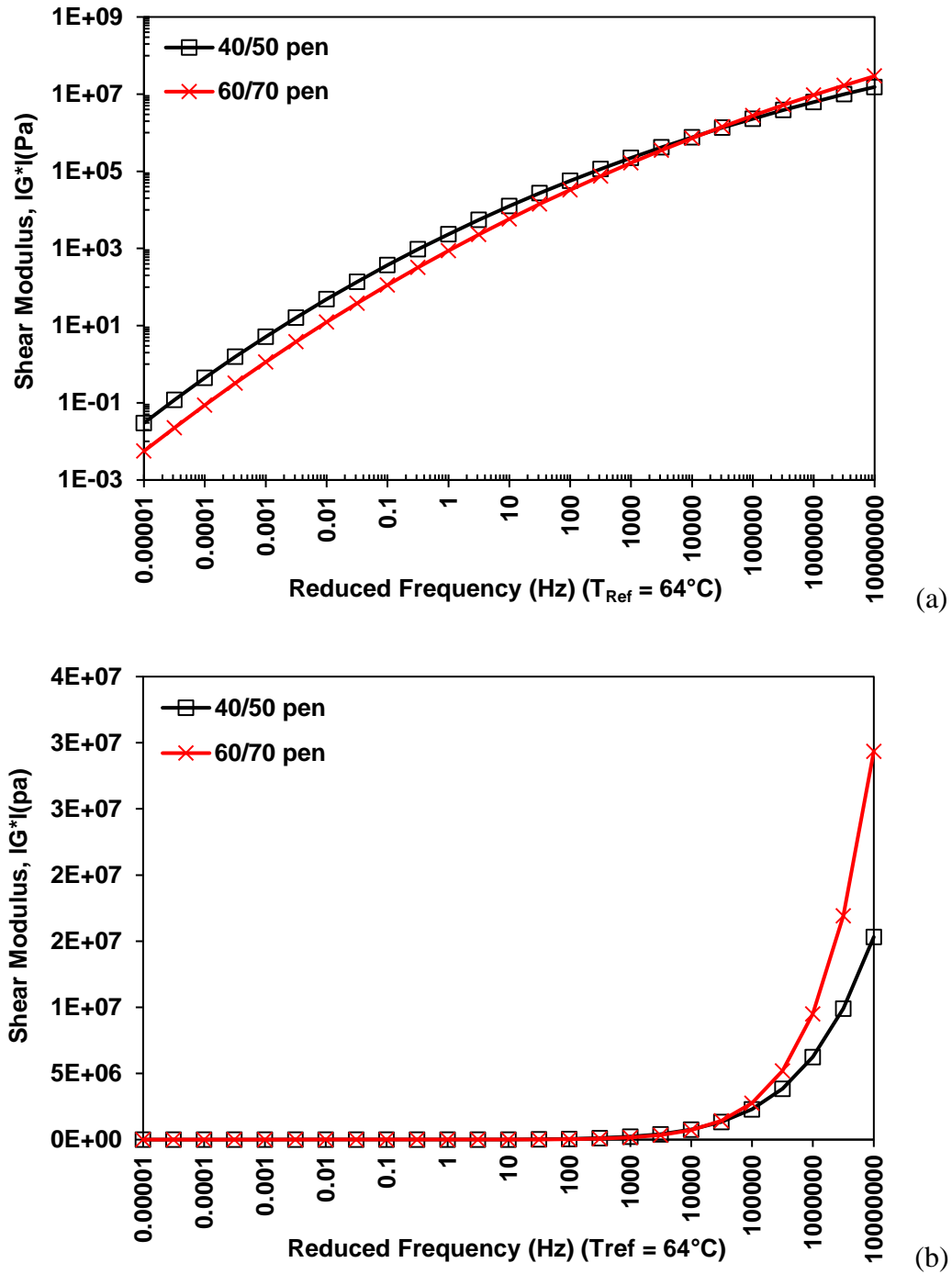
One of the primary analytical techniques used in analyzing the dynamic mechanical data for the SHRP asphalts involved construction of master curves for the dynamic complex modulus and



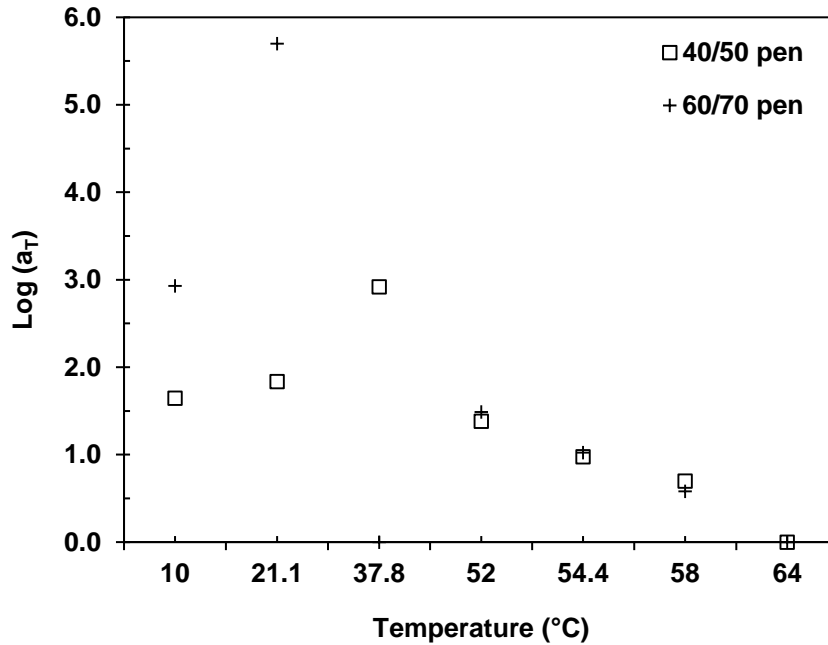
phase angle. In constructing such master curves, the TTS principle, or method of reduced variables, is used. In constructing a master curve using TTS, dynamic data are first collected over a range of temperatures and frequencies. A standard reference temperature must then be selected [10]. In this study 64°C was used as the reference temperature for constructing master curves. The data at all other temperatures are then shifted with respect to time until the curves merge into a single smooth function. The amount of shifting required at each temperature to form the master curve is of special importance, and is called the shift factor,  $a_T$ . A plot of  $\log a_T$  versus temperature is generally prepared in conjunction with the master curve. This type of plot gives a visual indication of how the properties of a viscoelastic material change with temperature. The time or frequency scale used in a master curve is referred to as reduced time or reduced frequency. The time-temperature superposition was done by simultaneously solving for the four coefficients of the sigmoidal function ( $\delta$ ,  $\alpha$ ,  $\beta$ , and  $\gamma$ ) as described in equation 20 and the two model coefficients ( $C_1$  and  $C_2$ ) as described in equation 21. The “Solver” function of the Microsoft Excel was used to conduct the nonlinear optimization for simultaneously solving these parameters for each of the asphalt binders.

Figure 31 presents the shear modulus master curves of the studied asphalt binders using log-log and semi-log scales. The log-log representation of the master curves can be used to assess the stiffness properties of asphalt binders tested at intermediate and higher temperatures and the low temperature stiffness properties can be evaluated using master curves plotted on semi-log scale. The test temperature shift factors for the asphalt binders evaluated in this study are shown in Figure 32. From Figure 31a it is observed that, at low and intermediate reduced frequency 40/50 pen resulted in higher stiffness properties. At low test temperature and high reduced frequency

60/70 pen showed higher stiffness properties (Figure 31b). Overall, as the reduced frequency increases the shear modulus increases.



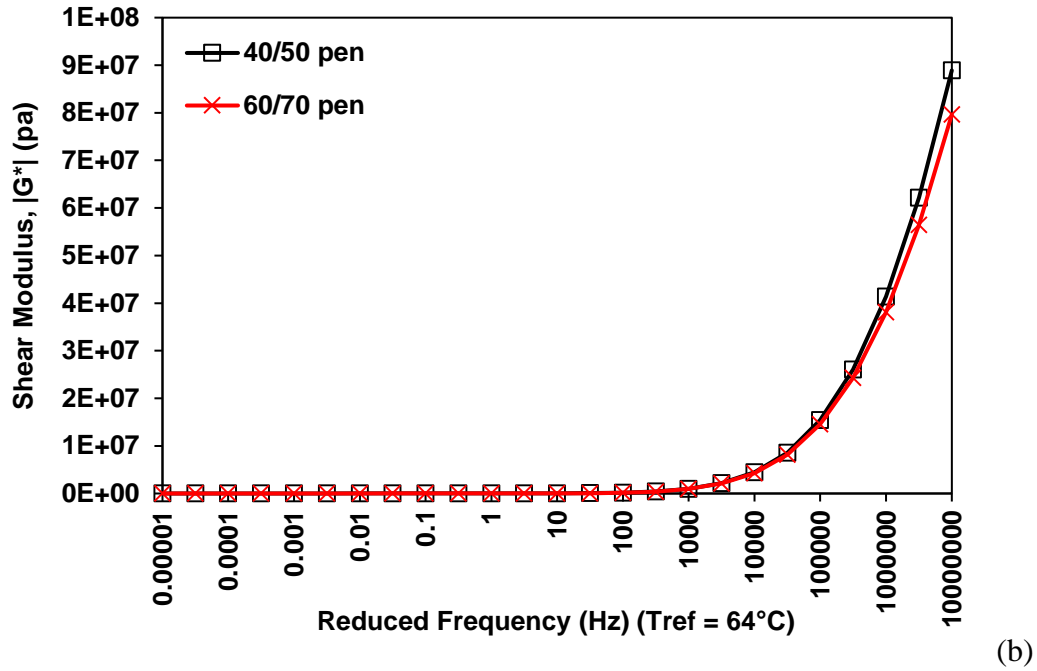
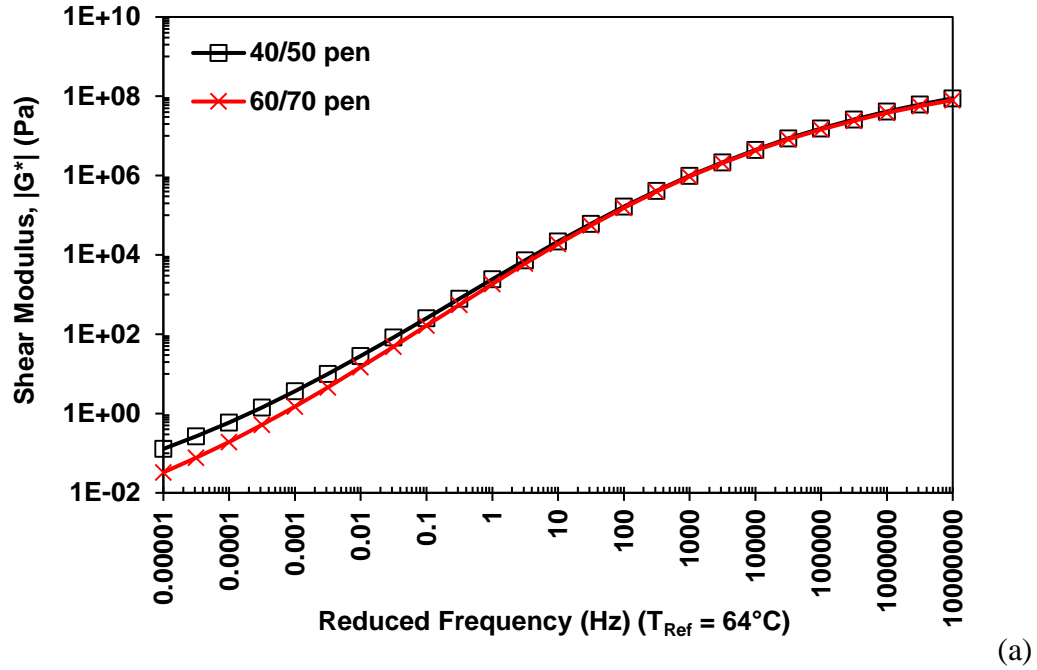
**Figure 31.** Shear modulus master curve; scale (a) log-log and (b) semi-log



**Figure 32.** *Master curve shift factors*

#### **4.9. Master Curve using FST Test Result**

Figure 33 presents the shear modulus master curves using log-log and semi-log scale for the studied asphalt binders. From Figure 33a it is observed that, at low reduced frequency 40/50 pen resulted in higher stiffness properties. When the low and intermediate test temperatures are considered 40/50 pen showed higher stiffness properties (Figure 33b). However, the test binders exhibited similar viscoelastic properties for wide range of reduced frequencies. Overall, as the reduced frequency decreases the shear modulus decreases.



**Figure 33.** Shear modulus master curves; scale (a) log-log and (b) semi-log

#### 4.10. MSCR and FST Master Curve Comparison

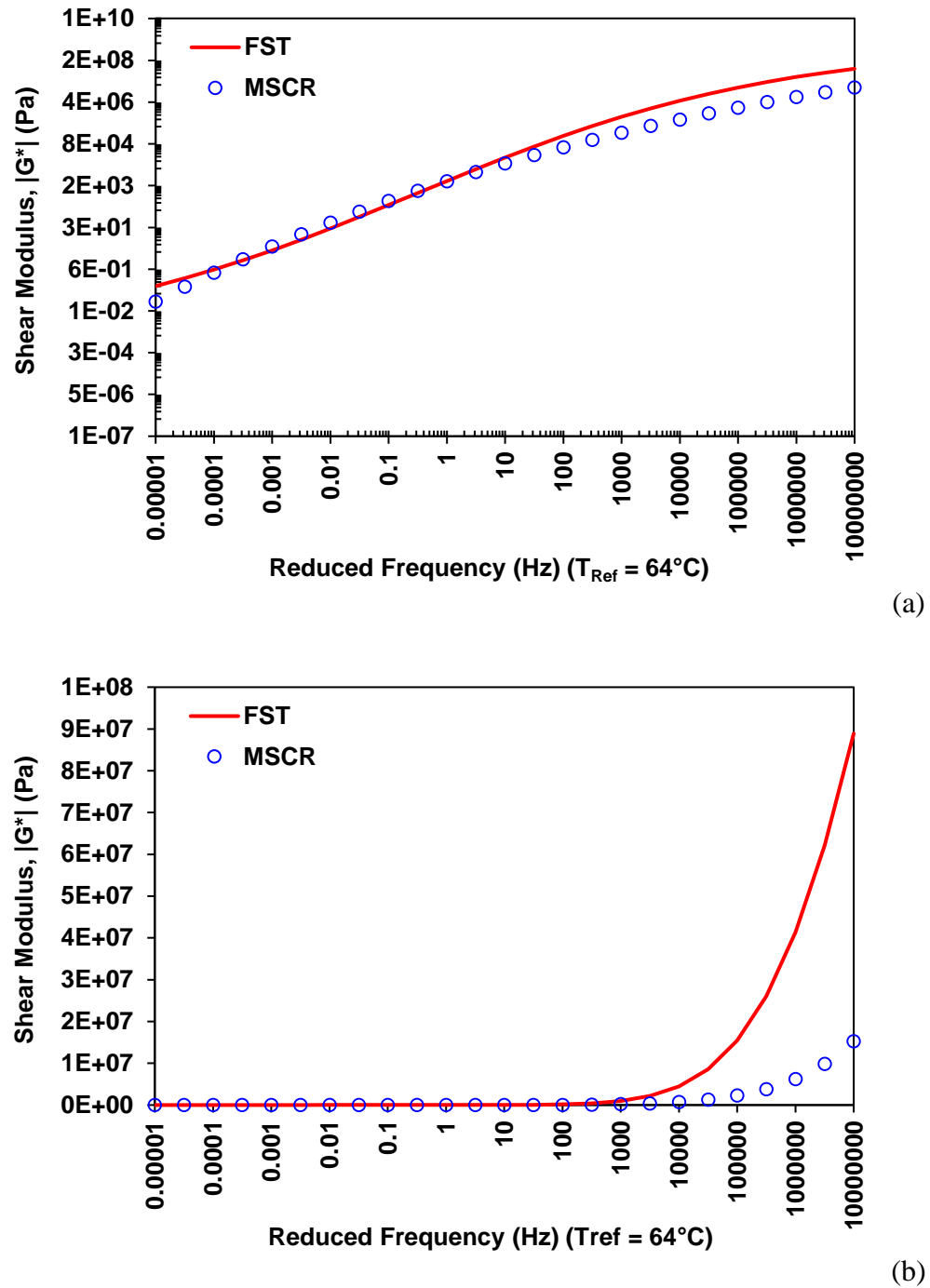
The aim of this paper is to estimate master curve results corresponding to the MSCR test temperatures and evaluate the accuracy of the predicted master curve properties at high

temperature and lower frequency ranges. The shear modulus master curves presented in Figure 31 were estimated based on material properties obtained from MSCR tests. To obtain material properties at extremely high frequency ranges, test results from low temperature range are required. For this purpose the MSCR estimated master curves were compared with master curves obtained from the FSTs. The FST data were obtained from tests carried out for temperatures ranging from 10 to 54.4 °C. To allow comparison with the MSCR estimations, the FST master curves were generated at a reference temperature of 64 °C.

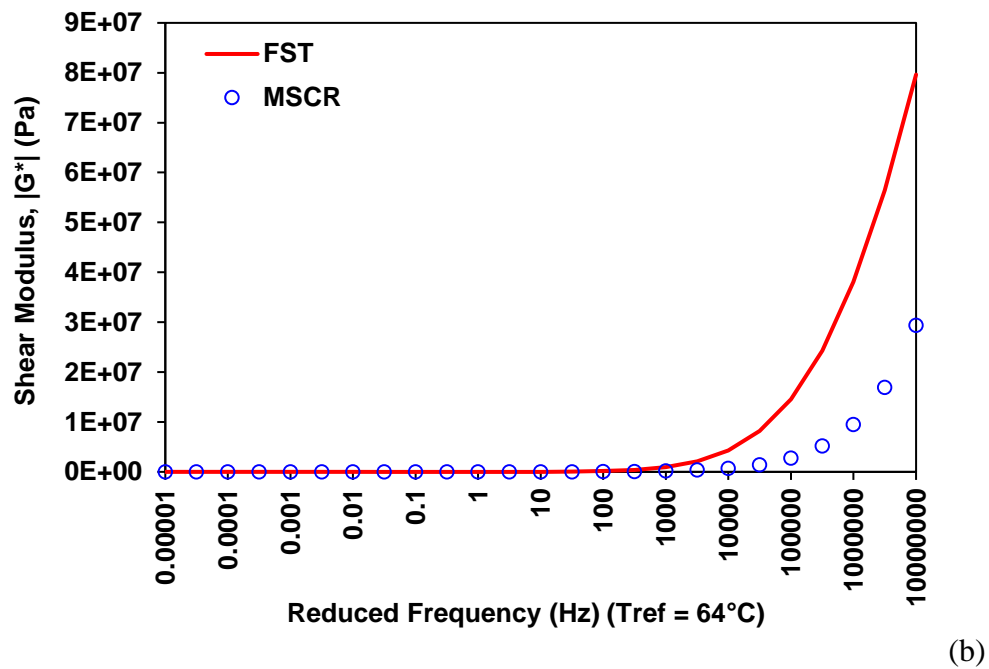
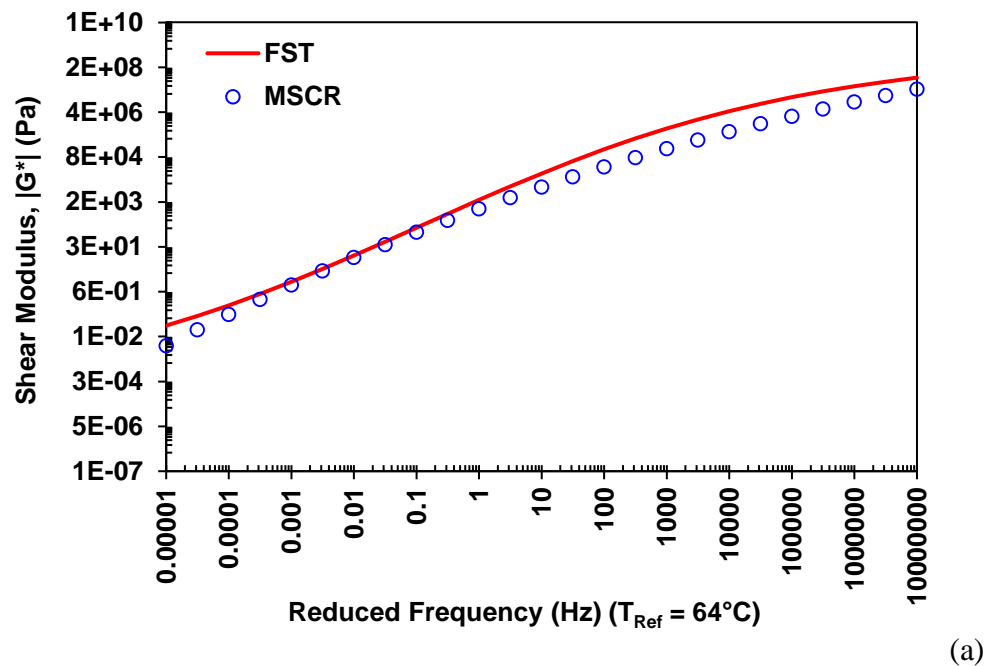
Figure 34 and 35 shows comparison of the shear modulus master curves derived from MSCR for temperatures ranging from 10°C to 64°C and FST of the tested asphalt binder at a reference temperature of 64 °C using log-log and semi-log scale. Similar trend was observed when the low and intermediate test temperature is considered (Figure 34a & 35a). Overall, the MSCR master curves satisfactorily matched the FST master curves at lower reduced frequency which corresponds to the high temperature ranges. Reasonably better agreement was obtained for 40/50 pen asphalt binders.

Comparison of the shear modulus master curves derived from MSCR for temperatures ranging from 52°C to 64°C and FST of the studied asphalt binder at a reference temperature of 64 °C is presented in Figure 36 & 37. Better agreement was observed when the intermediate temperature is considered (Figure 36a and 37a). Overall, the MSCR master curves satisfactorily matched the FST master curves at intermediate reduced frequency which corresponds to the intermediate temperature ranges. Reasonably better agreement was obtained for 40/50 pen asphalt binders. It is important to note that the estimation of viscoelastic properties of the MSCR measurements did not include low temperature and therefore a significant variation in the stiffness master curves was observed at the high reduced frequency ranges (Figure 36b and 37b). On the contrary, it is

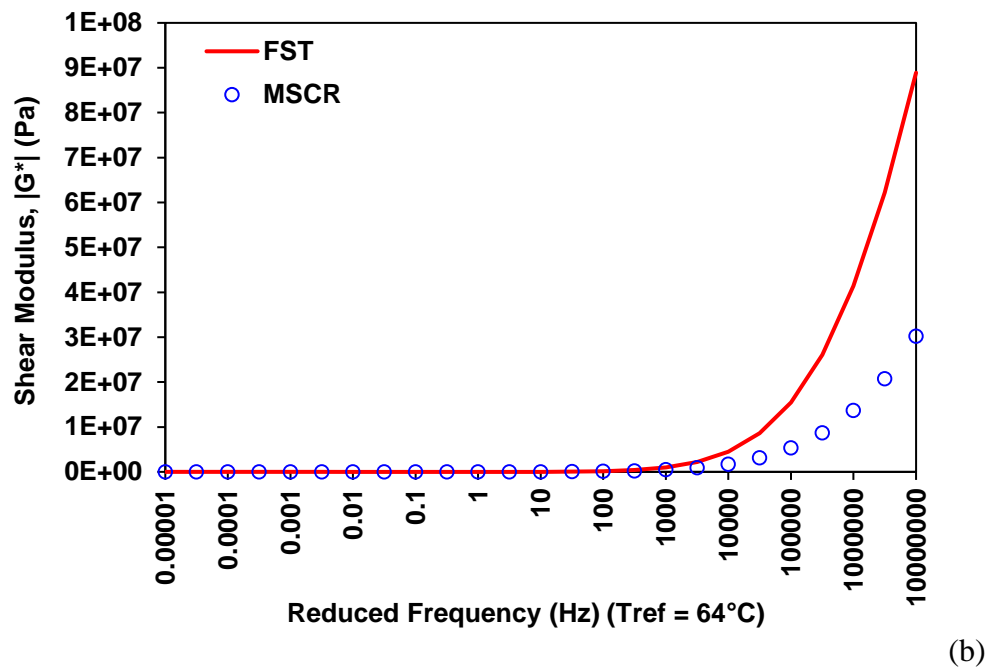
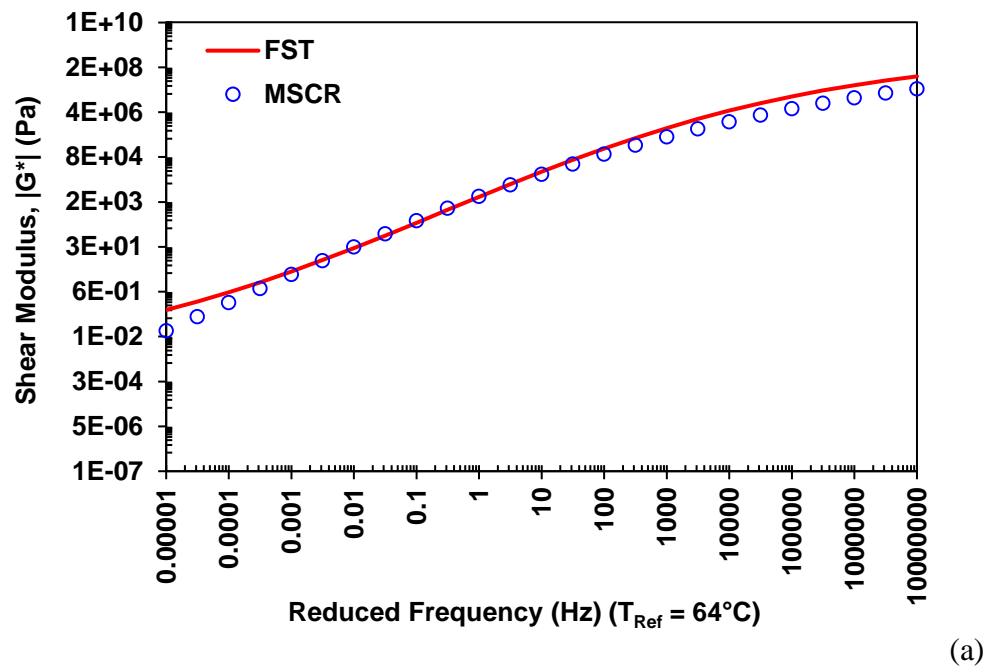
practically challenging to accurately estimate the glassy stiffness properties of the asphalt binders using high temperature MSCR measurements.



**Figure 34.** Comparison of master curve for 40/50 pen; scale (a) log-log and (b) semi log (Temperature =  $10^\circ\text{C}$  -  $64^\circ\text{C}$ )

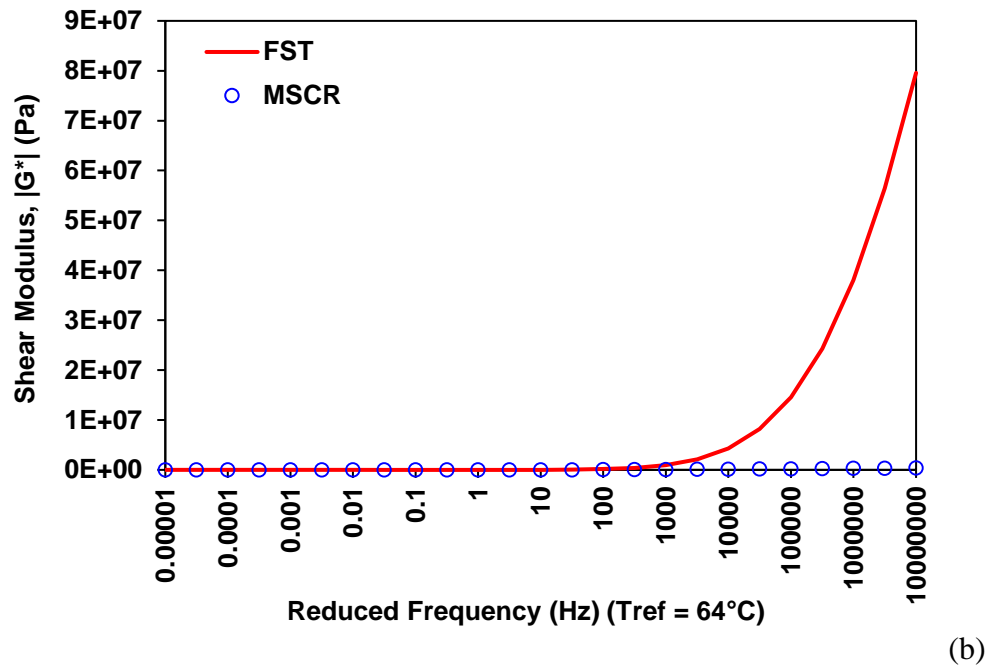
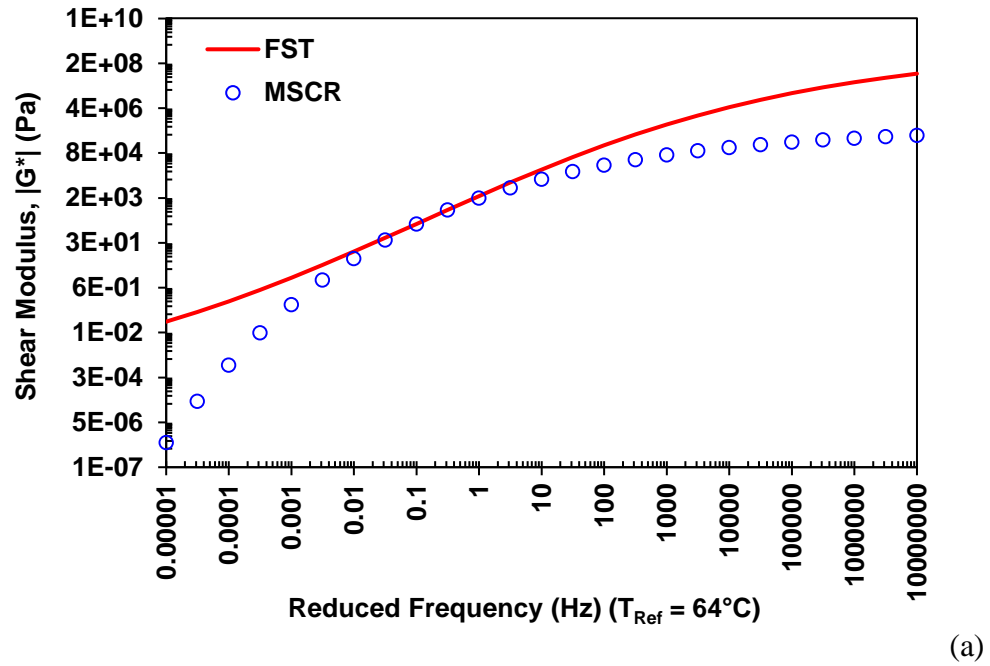


**Figure 35.** Comparison of master curve for 60/70 pen; scale (a) log-log and (b) semi-log  
(Temperature =  $10^\circ\text{C}$  -  $64^\circ\text{C}$ )



**Figure 36.** Comparison of master curve for 40/50 pen; scale (a) log-log and (b) semi-log  
(Temperature = 52°C - 64°C)





**Figure 37.** Comparison of master curve for 60/70 pen; scale (a) log-log and (b) semi-log  
(Temperature =  $52^\circ C - 64^\circ C$ )

Overall, the remarkably good agreement between the MSCR and FST master curves (Figure 34 & 35) demonstrates the possibility of obtaining overall stiffness properties of asphalt binders on

the basis of only MSCR test data. The findings also proves that the viscoelastic properties of the asphalt binders are essential and therefore material characteristics determined from MSCR can directly or indirectly be related to the results obtained from FST.

## CHAPTER 5

### 5. CONCLUSION AND RECOMMENDATION

#### 5.1. Conclusion

This paper presents evaluation of two asphalt binders which are 40/50 pen and 60/70 pen. The rheological properties of these binders were characterized using the oscillatory FST and MSCR test methods. Based on the laboratory test results and the analysis made in this paper, the following conclusions can be obtained:

1. The results of the conventional tests carried out for the two asphalt binders showed that all results are within the AASHTO specification limit.
2. The SuperPave PG grading of the two asphalt binder resulted in PG 64-yy which means the binders have adequate stiffness properties to withstand a pavement temperature of at least 64°C.
3. 60/70 pen asphalt binder resulted in higher  $J_{nr}$  and lower R values as compared to 40/50 pen. It is also observed that as the test temperature increases,  $J_{nr}$  increases and R decreases for both binders.
4. Higher values of  $G_1$  and  $G_2$  in conjunction with higher values of  $\eta_1$  and  $\eta_2$  were observed for 40/50 pen which is an indicator of better stiffness and higher resistance to deformation.
5. The estimated shear strain values satisfactorily matches the measured shear strain values for both binders and higher deformation was recorded for 60/70 pen. The statistical analysis which was conducted to indicate the goodness-of-fit between the measured and the estimated shear strain values showed that excellent estimation of the creep and recovery strains.

6. The statistical analysis which was performed to indicate the goodness-of-fit between the measured and the estimated creep and recovery properties resulted in acceptable estimate of the creep and recovery of the creep and recovery properties.
7. Similar viscoelastic properties were observed for both binders at low, intermediate and high frequency. However, faster transition time was observed for 40/50 pen.
8. The shear modulus master curves derived from MSCR and FST test methods were compared and remarkably good agreement was obtained.

It is also observed that the 40/50 pen asphalt binder resulted in higher viscoelastic properties and resulted in higher resistance to shear deformation as compared to the 60/70 pen asphalt binder. Based on the statistical analysis which was conducted to objectively evaluate the accuracy of the estimated viscoelastic properties, the Burgers model parameters were found to be suitable and adequate for describing the creep and recovery viscoelastic properties of the asphalt binders included in the study.

## **5.2. Recommendation**

Based on the final results obtained from this study, it is recommended to implement MSCR test over FST because:

- The MSCR specification (M 332) could be implemented and applied to all binders regardless of modification.
- The MSCR test grades the binders considering both environmental and traffic conditions. That is, the expected traffic levels do not have to be addressed by so-called “grade bumping”.
- In addition, the MSCR test is expected to optimize the binder formulation to avoid the use of over engineered binders.

- MSCR test is a simple, quick and economical test method to characterize the viscoelastic properties of asphalt binder with respect to repeated creep loading response.

Finally I would like to recommend Ethiopian highway agencies to implement SuperPave PG specification because the rheological tests under this system are performance related so the results from such tests better correlate with the field conditions.

### **5.3. Future Study**

Future study may include evaluating the proposed approach using modified asphalt binders tested at a wide range of temperatures. And also, the study may evaluate the nonlinear viscoelastic properties of the asphalt binders derived from MSCR measurement.

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## APPENDICES

### APPENDIX A - Conventional Test Result

**Table A-1** *Summary of Conventional Test for 40/50pen asphalt binder*

Test	Result	ASTM Specification Limit
Penetration (mm)	45.33	40-50
Ductility (cm)	100.33	Min 100
Softening Point (°C)	56	52-60
Flash and Fire Point (°C)	316	Min 250

**Table A-2** *Summary of Conventional Test for 60/70 pen asphalt binder*

Test	Result	ASTM Specification Limit
Penetration (mm)	64.67	60-70
Ductility (cm)	100	Min 100
Softening Point (°C)	52	49-56
Flash and Fire Point (°C)	321	Min 250

## APPENDIX B – Performance Grade (PG) Test Result

**Table B-1** *Performance Grade determination of 40/50 pen RTFO aged asphalt binder*

Temperature (°C)	Frequency (Hz)	Phase Angle (°)	Complex Modulus (Pa)	Elastic Modulus (Pa)	Viscous Modulus (Pa)	Complex Viscosity (Pas)	Shear Stress (Pa)	Strain ()
63.94	1.60E+00	84.03	5.02E+03	5.23E+02	5.00E+03	5.01E+02	5.03E+02	1.00E-01
69.93	1.60E+00	85.8	2.47E+03	1.81E+02	2.46E+03	2.46E+02	2.47E+02	1.00E-01
75.9	1.60E+00	87.14	1.13E+03	5.65E+01	1.13E+03	1.13E+02	1.16E+02	1.02E-01

**Table B-2** *Performance Grade determination of 60/70 pen RTFO aged asphalt binder*

Temperature (°C)	Frequency (Hz)	Phase Angle (°)	Complex Modulus (Pa)	Elastic Modulus (Pa)	Viscous Modulus (Pa)	Complex Viscosity (Pas)	Shear Stress (Pa)	Strain ()
64.14	1.60E+00	87.54	1.38E+03	5.91E+01	1.37E+03	1.37E+02	1.37E+02	9.98E-02
69.9	1.60E+00	88.34	6.74E+02	1.95E+01	6.74E+02	6.72E+01	6.72E+01	9.97E-02

## APPENDIX C – Creep and Recovery Properties

**Table C-1** *Summary of  $J_{nr}$  and  $R$  results at 100 Pa*

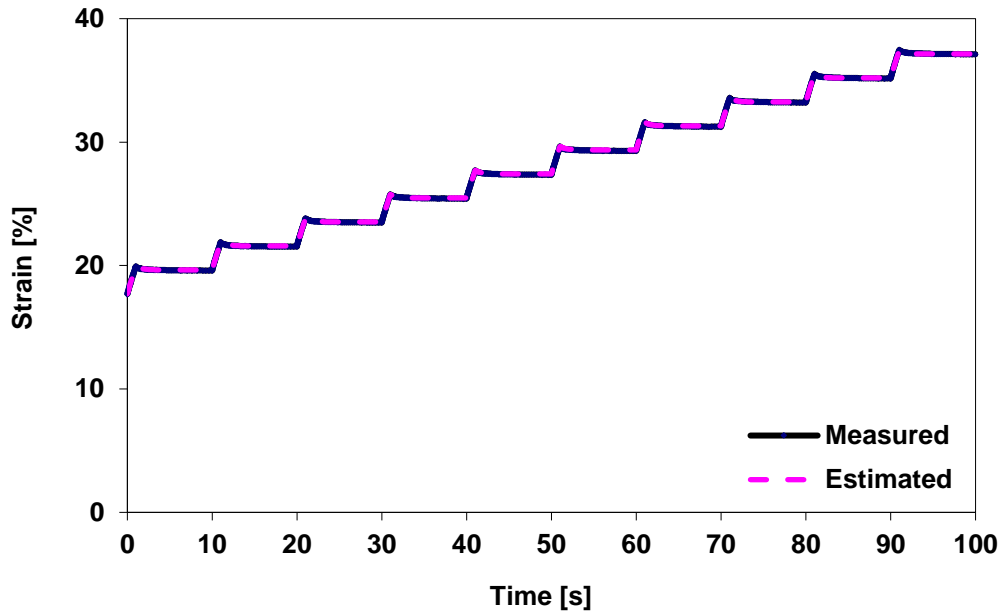
<b>Binder ID</b>	<b>Temperature (°C)</b>	<b><math>J_{nr}</math> (1/kPa)</b>	<b><math>R</math> (%)</b>
40/50 pen	52	0.19	15.47
	58	0.51	9.77
	64	2.04	5.38
60/70 pen	52	0.13	30.63
	58	1.74	3.83
	64	4.24	3.30

## APPENDIX D – Burger Model Parameters

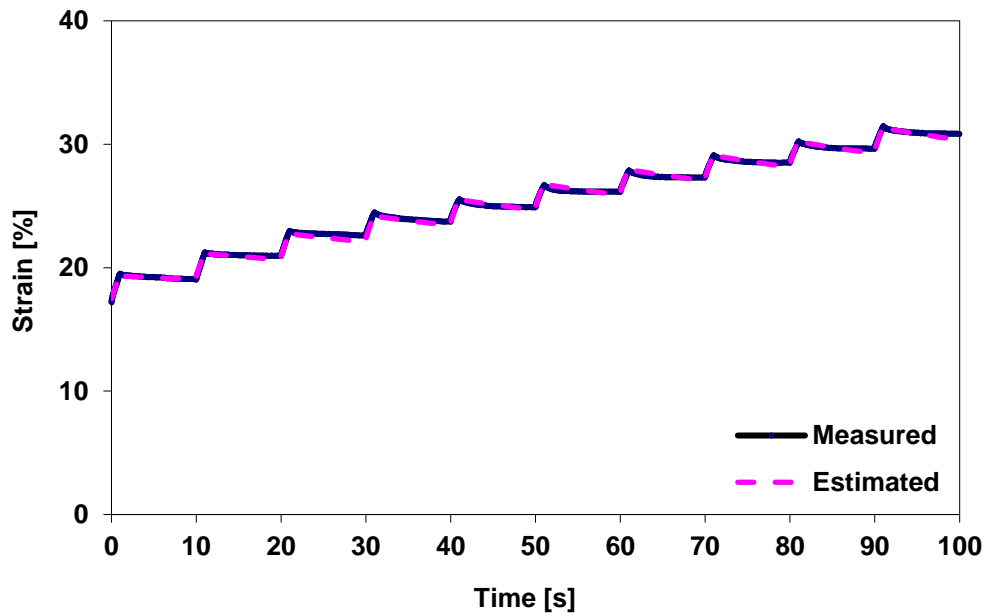
**Table D-1** *Summary of Burger Model Parameters results at 100 Pa*

<b>Binder ID</b>	<b>Temperature (°C)</b>	<b>G<sub>1</sub></b>	<b>η<sub>1</sub></b>	<b>G<sub>2</sub></b>	<b>η<sub>2</sub></b>
40/50 pen	52	3.55E+05	5.20E+03	2.65E+04	1.89E+04
	58	3.54E+04	1.91E+03	3.54E+01	1.28E+07
	64	1.72E+04	2.36E+04	1.56E-13	4.97E+02
60/70 pen	52	3.26E+04	9.59E+03	3.18E+02	9.21E+03
	58	1.54E+04	7.28E+03	1.56E-13	6.22E+02
	64	9.23E+03	8.36E+03	1.56E-13	2.42E+02

## APPENDIX E – Measured and Estimated Strain



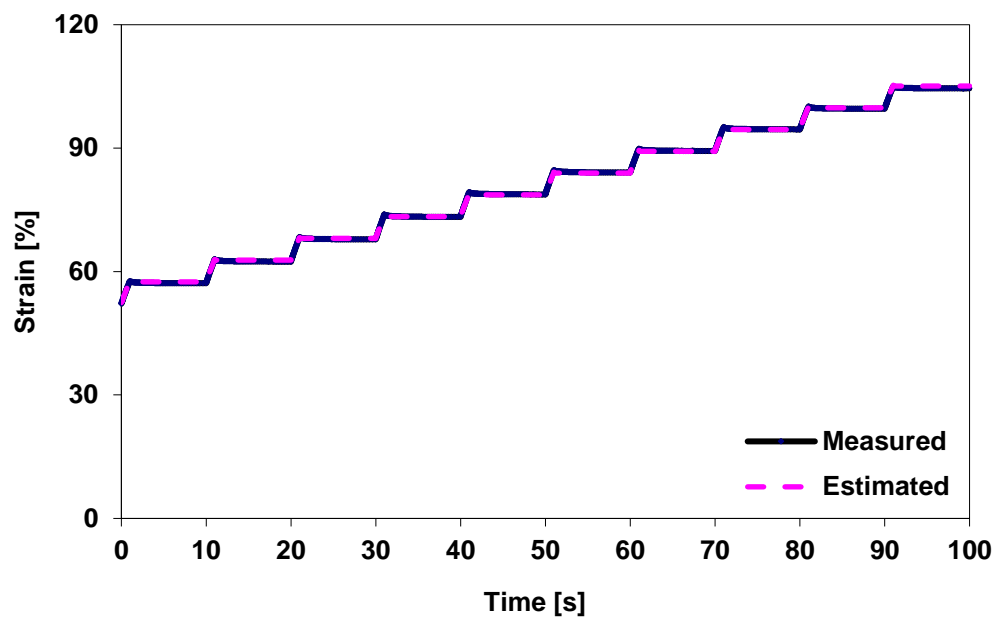
(a)



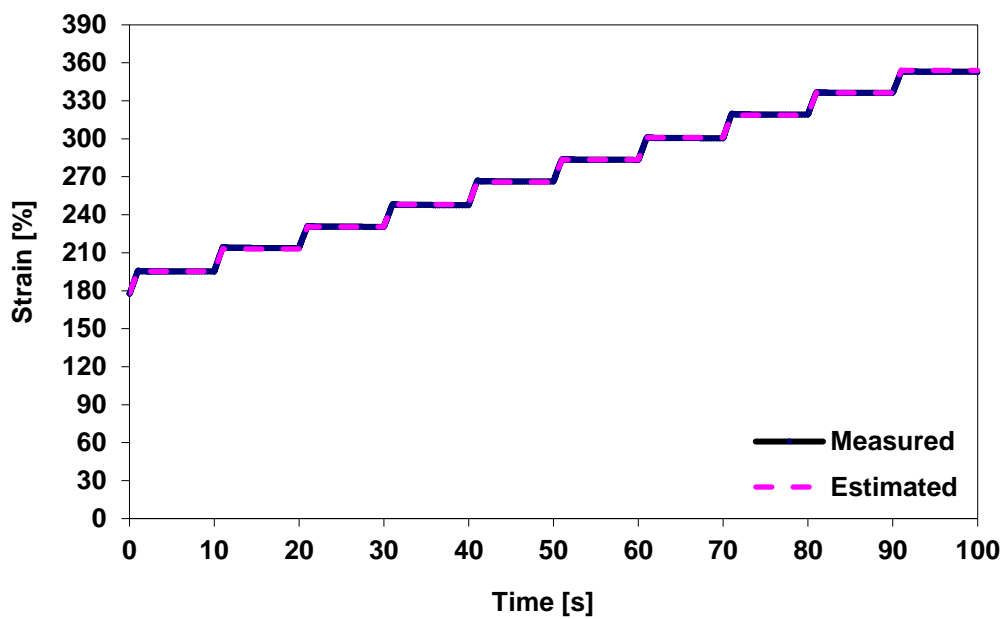
(b)

**Figure E-1** Comparison of Measured and Estimated Shear Strain at 100 Pa and 52°C

(a) 40/50 pen and (b) 60/70 pen



(a)



(b)

**FigureE-2** Comparison of Measured and Estimated Shear Strain at 100 Pa and 58°C

(a) 40/50 pen and (b) 60/70 pen

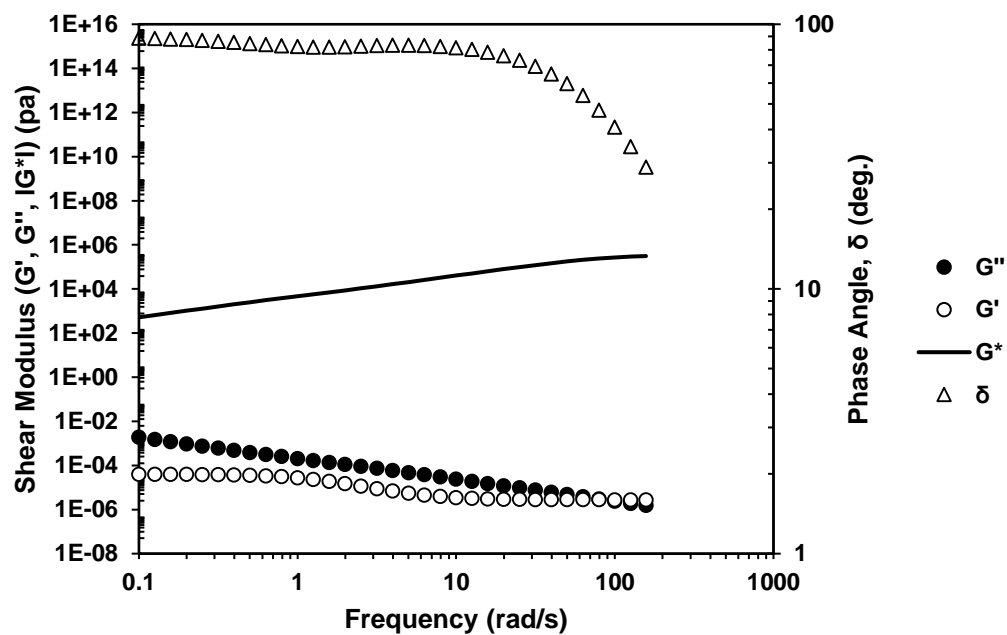


## APPENDIX F – Measured and Estimated Creep and Recovery Properties

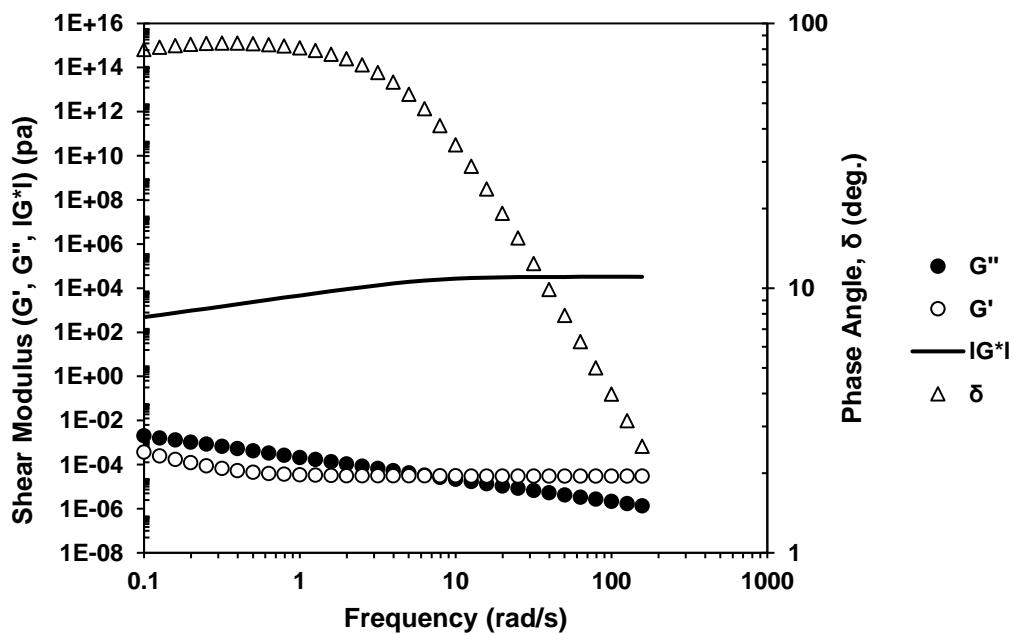
**Table F-1** *Comparison of Measured and Estimated  $J_{nr}$  and  $R$  results at 100 Pa*

Binder ID	Temperature (°C)	J <sub>nr</sub> (1/kPa)		R (%)	
		Measured	Estimated	Measured	Estimated
40/50 pen	52	0.19	0.19	15.47	12.63
	58	0.51	0.50	9.77	3.50
	64	2.04	2.01	5.38	0.89
60/70 pen	52	0.13	0.11	30.63	50.87
	58	1.73	1.69	4.30	1.81
	64	4.24	4.17	3.30	0.61

## APPENDIX G – Viscoelastic Properties



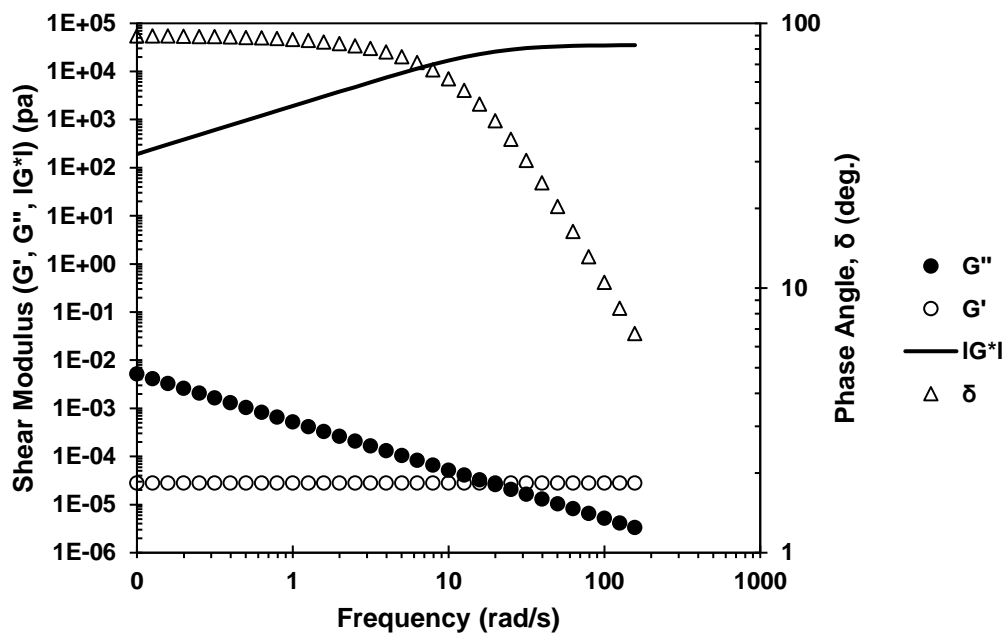
(a)



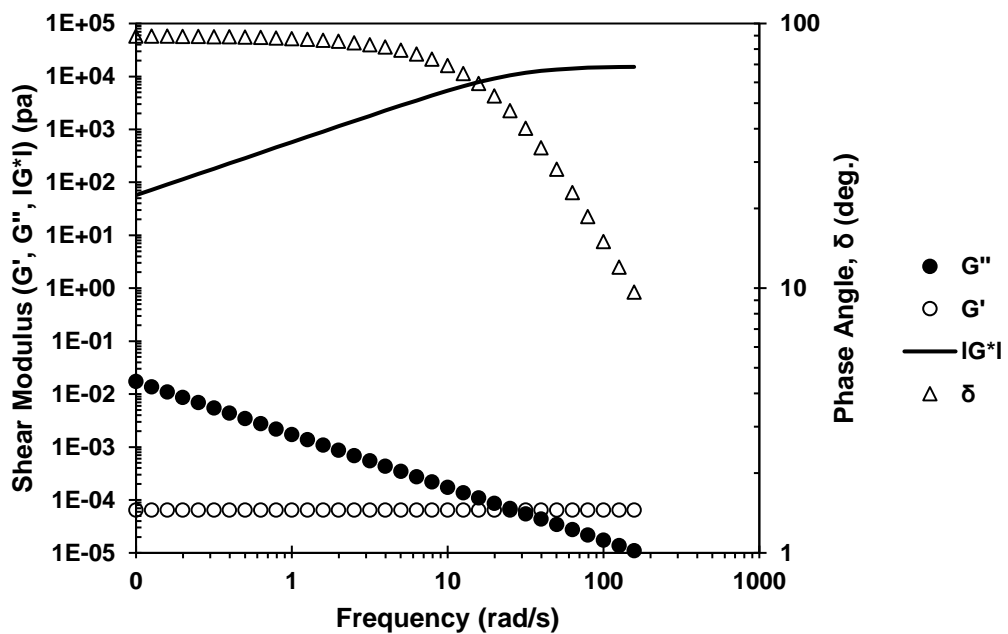
(b)

**Figure G-1** Comparison of Viscoelastic Properties at 100 Pa and 52°C

(a) 40/50 pen and (b) 60/70 pen



(a)



(b)

**Figure G-2** Comparison of Viscoelastic Properties at 100 Pa and 58°C

(a) 40/50 pen and (b) 60/70 pen